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DRL NO. 81
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DOE/JPL 954334 – 17
Distribution Category UC - 63

QUARTERLY PROGRESS REPORT

OCT – DEC 1980

low cost solar array project

EXPERIMENTAL PROCESS SYSTEM DEVELOPMENT UNIT FOR PRODUCING SEMICONDUCTOR-GRADE SILICON USING THE SILANE-TO-SILICON PROCESS

UNION CARBIDE CORPORATION

THE JPL LOW-COST SOLAR ARRAY PROJECT IS SPONSORED BY THE U.S. DEPARTMENT OF ENERGY AND FORMS PART OF THE SOLAR PHOTOVOLTAIC CONVERSION PROGRAM TO INITIATE A MAJOR EFFORT TOWARD THE DEVELOPMENT OF LOW-COST SOLAR ARRAYS. THIS WORK WAS PERFORMED FOR THE JET PROPULSION LABORATORY, CALIFORNIA INSTITUTE OF TECHNOLOGY BY AGREEMENT BETWEEN NASA AND DOE.

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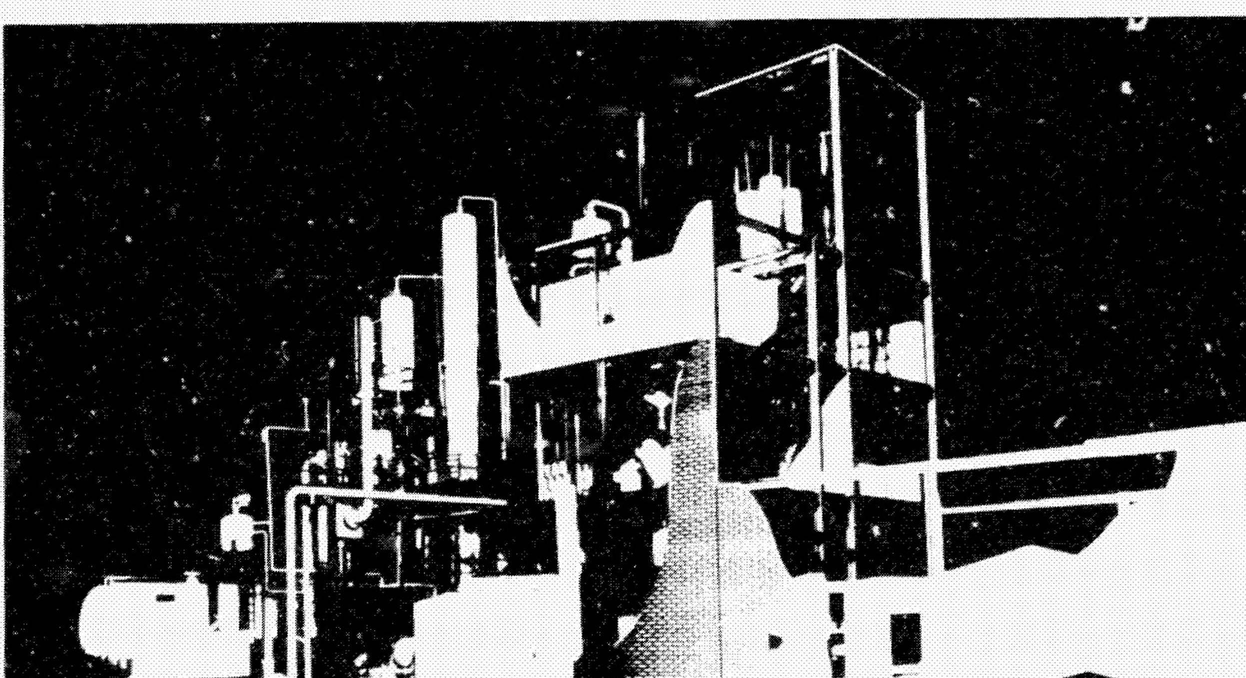

UNION CARBIDE CORPORATION

TABLE OF CONTENTS

TABLE OF CONTENTS (continued)

TABLE OF CONTENTS (continued)

TABLE OF CONTENTS (continued)

TABLES

TABLE OF CONTENTS (continued)

ILLUSTRATIONS

ABSTRACT

This report covers work performed in October, November, and December, 1980 on JPL/DOE Contract 954334, Phase III. This phase consists of the engineering design, fabrication, assembly, operation, economic analysis, and process support R&D for an Experimental Process System Development Unit (EPSDU).

The EPSDU facility design is converging to bring together the process, civil, mechanical, electrical and equipment designs.

The "disinterested" safety review team met, and performed an extensive review of the EPSDU design which almost consumed a week. Comments and suggestions of the safety review team were incorporated into the subsequent P&I ·d the mechanical installation design reviews. Design changes necessitated ᴗy these reviews will be incorporated into the design drawings during the next quarter.

Most of the process related equipment has been ordered and is being fabricated. A large number of purchase orders were written in December for field instruments and controls. A total of 116 validated purchase orders have been issued. Equipment items have started to arrive at the EPSDU site.

Equipment and building foundations have been completed at the EPSDU site, and all the steel was erected for the gantry. The switch gear/control building and the melter building will be completed during the next quarter.

The data collection system design has picked up pace. Various computer programs are being written which will be used to convert electrical, pneumatic and other raw signals into engineering values. They are then stored, printed in a usable form, or entered into data reduction programs for study.

Two process R&D tasks have been successfully completed this quarter. The free-space reactor development work was completed with a final 12-hour run in which the free-space reactor PDU ran flawlessly. Also, the quality control method development task was completed. We were able to grow slim rods from seed silicon rods for subsequent float zone operation and impurity

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characterization. We also deposited an excellent quality epitaxial film on a silicon wafer. Both undoped and doped films were deposited and the resistivity of the films have been measured.

Kayex has assembled over 70 percent of the powder melter/shotter system. Some key equipment such as an induction generator should be delivered soon. Check-out and start-up should commence next quarter.

The fluid-bed pyrolysis of silane is an attractive alternative to the free-space reactor and the melter/shotter system. Its installation is nearing completion. The check-out and calibration will be done next quarter. An operating manual has been written.

Overall, the EPSDU engineering and R&D programs are progressing well. As we pointed out in the third quarter, preparation of P&I diagram and the mechanical installation drawings has been more time-consuming than originally planned. Substantial progress was made this quarter by assigning more manpower.

SECTION I. INTRODUCTION

This report covers work performed in October, November and December 1980 on JPL/DOE Contract 954334, Phase III.

The overall objective of the LSA Silicon Material Task is to establish a chemical process for producing silicon at a rate and price commensurate with the production goals of the LSA project for solar-cell modules. This material must be suitable for utilization in the large-area sheet process and in the automated process for the fabrication of solar cells having satisfactory physical and electrical performance characteristics.

As part of the overall Silicon Material Task, Union Carbide developed the silane-silicon process and advanced the technology to the point where it has a definite potential for providing high-purity polysilicon on a commercial scale at a price of $14/kg by 1986 (1980 dollars). This work, completed under Phases I and II of the contract, provided a firm base for the Phase III Program (initiated in April 1979) aimed at establishing the practicality of the process by pursuing the following specific objectives:

- Design, fabricate, install, and operate an Experimental Process System Development Unit (EPSDU) sized for 100 MT/Yr to obtain extensive performance data to establish the data base for the design of commercial facilities.

- Perform supporting research and development to provide an information base usable for the EPSDU and for technological design and economic analysis for potential scale-up of the process.

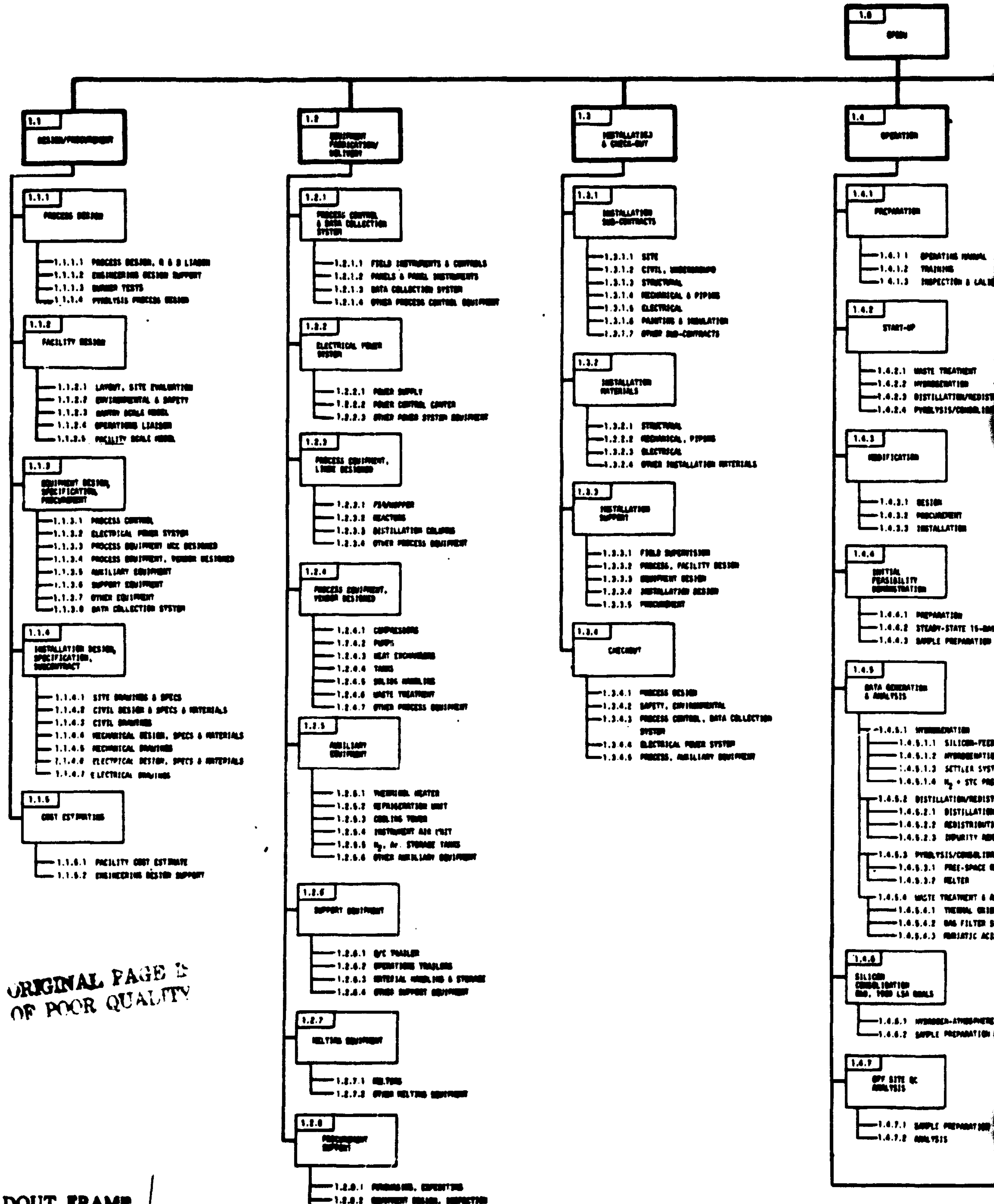
- Perform iterative economic analyses of the estimated product cost for the production of semiconductor-grade silicon in a facility capable of producing 1000 MT/Yr.

This process for preparing semiconductor-grade silicon in the EPSDU from metallurgical-grade (M-G) silicon is based on a well-integrated arrangement of purification steps that provides a cost-effective process system.

The three basic steps entail converting M-G silicon to trichlorosilane, redistributing the trichlorosilane to produce silane, and thermally decomposing the silane to form amorphous silicon powder. The powder is then melted and the molten silicon is cast into polycrystalline silicon for subsequent use in fabricating solar cells.

The technical progress presented in this report is arranged according to the Work Breakdown Structure (WBS) shown in Table I.

TABLE I WORK BREAKDOWN STRU



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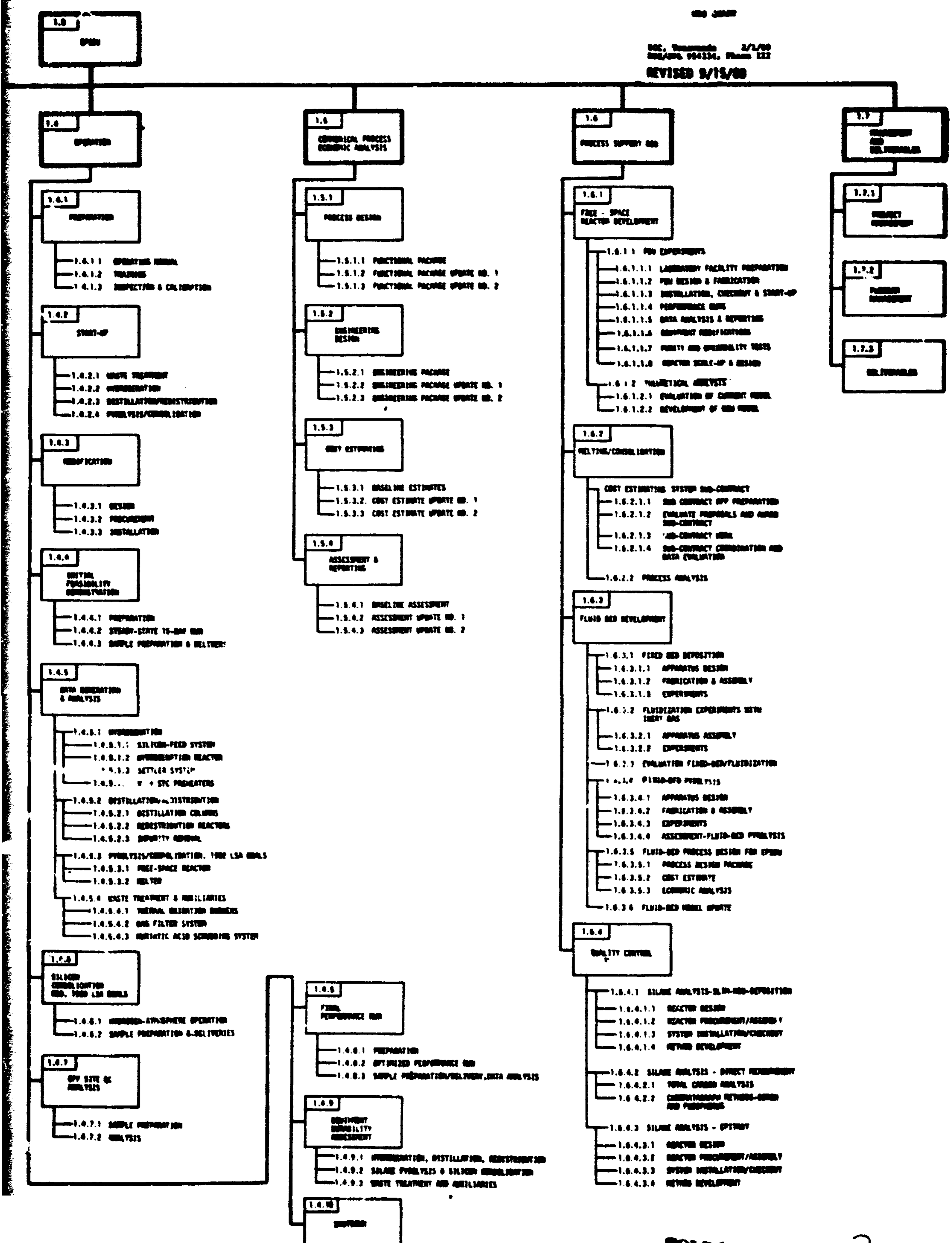

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SECTION II. TECHNICAL ACTIVITIES (BY WBS NUMBER)

1. EPSDU PROGRAM

As illustrated in Table I, the current phase III program consists of seven primary (WBS level 2) divisions of effort:

- EPSDU Design and Procurement
- EPSDU Equipment Fabrication and Delivery
- EPSDU Installation and Checkout
- EPSDU Operation
- Commercial (1000 MT/Yr) Process Economic Analysis
- Process R&D to Support EPSDU Design and Commercial Analysis
- Program Management

Collectively, these activities encompass all effort required to attain the program objectives. The subdivisions (WBS levels 3, 4, and 5) define the individual work items that must be performed. The progress for this quarter, documented in this section, is reported at the work-item level. Only items that are currently in work are included.

1.1 EPSDU DESIGN/PROCUREMENT

This effort includes all engineering, design, and procurement activities necessary to transform the process design, developed during the Phase II Program, into a complete installation-drawing package for EPSDU. The major tasks include process design updates, facility design, equipment design and procurement, installation design, and cost estimating support.

1.1.1 Process Design

The process design effort is geared toward using the most recent information available to provide the most practicable integration of process subsystems for attaining the EPSDU Program objectives. The process design package consists of a heat/mass balance, process description, process

flow diagram, and functional specifications for process equipment. The original package, issued in June 1979, served as the basis for the subsequent engineering effort. Beneficial data from the Supporting R&D effort and other process-related analyses and experiments were used to update the original package. Process engineers, using information available from the Phase I and Phase II Programs, provide direct support to the facility and equipment design efforts.

1.1.1.1 Process Design, R&D Liaison

The process design is continually being reviewed to identify refinements that have the potential for lowering costs, reducing requirements for specialized equipment, or alleviating problems that could arise during operation. The effort for this quarter was concentrated primarily on the folloiwng:

- Specification of Pressure Relief Valves
- P&I Drawing Review
- Mechanical Installation Drawing Review

1.1.1.2 Engineering Design Support

Because of the special process considerations required for EPSDU, process design personnel, using knowledge of the Phases I and II development effort and relevant chlorosilane work at other UCC locations, assist in the detail design of the facility and equipment. Although this participation encompasses many design activities, the effort for this period was primarily involved with reviewing the remaining vendor drawings, valve selection, component cleanliness requirements and safety items.

Valves

Effort was expended on the selection and sizing of the various small valves such as packless valves and check valves. There are approximately 200 such valves which were required to be screened for capability, availability and cost. Pressure relief valves were sized and added to the installation drawings.

Vendor Drawings

Vendor drawings are continuing to be reviewed as they are received.

Cleaning Standards

Some of our vendors are having difficulty meeting our electronic cleaning standards because they are unfamiliar with the applicable specifications. Liaison with vendors to tailor the specifications to fit specific equipment was initiated. This effort assures us of obtaining properly cleaned equipment at a reasonable cleaning cost.

Equipment cleaning requirements were reviewed. The components which cannot be cleaned by vendors will be cleaned by Union Carbide in accordance with SCS-1 specifications. Plans are being made to clean instruments and other sensitive items at Tonawanda and other items such as pipes will be cleaned on-site at East Chicago.

Mechanical Engineering/Installation Design

The drawings were reviewed for compatibility with the P&I diagram. The review was conducted line-by-line according to the latest P&I diagrams and the recommended changes are being incorporated in the P&I diagrams, installation design drawings and the scale model. A construction bid package incorporating these changes should be completed in April. Any further necessary changes will be resolved as field changes during construction. The pyrolysis/consolidation area and the QC trailer area will not be reviewed until their engineering design becomes firm.

Safety

Time was spent thoroughly reviewing the engineering design from operability and safety points of view. The design was examined for adequate provisions for start-up, emergency shut down, and draining of process fluids. Major and most minor problems were identified so that costly field changes will be avoided.

Gas Analysis

A set of data for vapor-liquid equilibrium on a 1% diborane in silane system was received. It was analyzed to insure that the silane

column reflux controls will have the correct range. This analysis was performed by re-running the distillation simulation program with the activity coefficients that resulted from the data. The number of trays will be constant and the product purity can be compared to the reflux.

1.1.1.4 Pyrolysis/Consolidation Process Design

Activity was delayed due to manpower requirements elsewhere, however, the work will restart in the next quarter.

1.1.2 Facility Design

Facility design consists primarily of the effort required to translate the process design functional requirements into specific plans regarding site, physical arrangement, human factors, and safety and environmental considerations. Personnel who will operate the facility participate to provide human factor inputs and to become familiar with the process.

1.1.2.2 Environmental and Safety

The design review team met in Tonawanda on November 18-20 with Dr. Hsu and Mr. Josephs representing JPL. The team consists of the following personnel who offer a wide range of design and operating experience within Union Carbide:

- R. M. Long - EPSDU Plant Manager
  Linde Division
  East Chicago, Indiana
- A. L. Altman - Process Design Engineer
  Engineering and Hydrocarbons Division
  South Charleston, West Virginia
- K. R. Bowman - Operations Design Engineer
  Silicones and Urethane Intermediates Division
  Sistersville, West Virginia
- R. S. Gacek - Control Systems Engineer
  Linde Division
  Tonawanda, New York
- G. Tarancon - R&D Chemist
  Specialty Gases/Linde Division
  Keasbey, New Jersey
- D. G. Keierleber - Operations Superintendent
  Specialty Gases/Linde Division
  Keasbey, New Jersey

W. C. Breneman and G. Hsu acted as ex-offico team members. Mr. Breneman coordinated the review sessions. The review included safety and operability of the M-G silicon-to-silane portion of the process and features of the EPSDU site layout, process equipment, and fire protection. The safety team comments were subsequently reviewed by engineering personnel and many of the suggested items are being implemented and the others will receive additional study in January.

1.1.2.3 Scale Model

The gantry scale model (Figure 1) was completed in October and was used to assist mechanical designers during the drawing checking effort.

The scale model will require revisions to piping arrangements as a result of the outcome of detailed design review meetings in early December. Revisions will be completed next year before the start of the mechanical installation.

1.1.2.4 Operations Liaison

The EPSDU plant manager (Mr. Long) attended program orientation and EPSDU design review meetings at Tonawanda. He will continue to be resident at East Chicago and will visit Tonawanda and other locations on approximately 2-week intervals to become familiar with the process, controls, operational know-how, and safety.

He also visited the Sistersville Silicones production facility for operations orientation, participated in the safety review meeting and in engineering/installation design reviews.

Detailed planning for the pre-startup activities for Operation (1.4) progressed substantially in December. Day-to-day coordination of the field construction activities at East Chicago continues.

1.1.2.5 Facility Scale Model

The facility scale model equipment has been fabricated and will be positioned based on the equipment location plan shown in Figure 2.

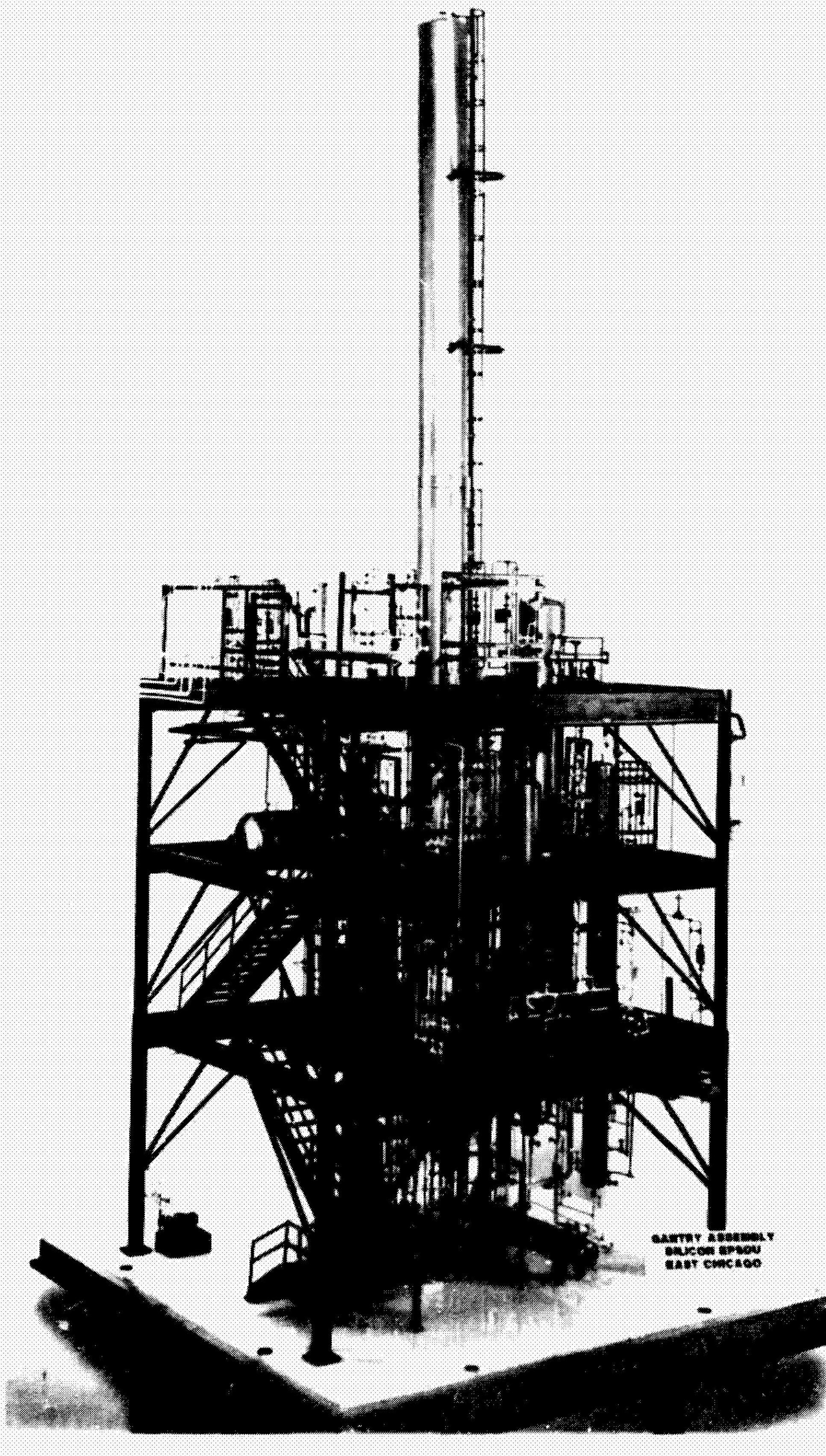
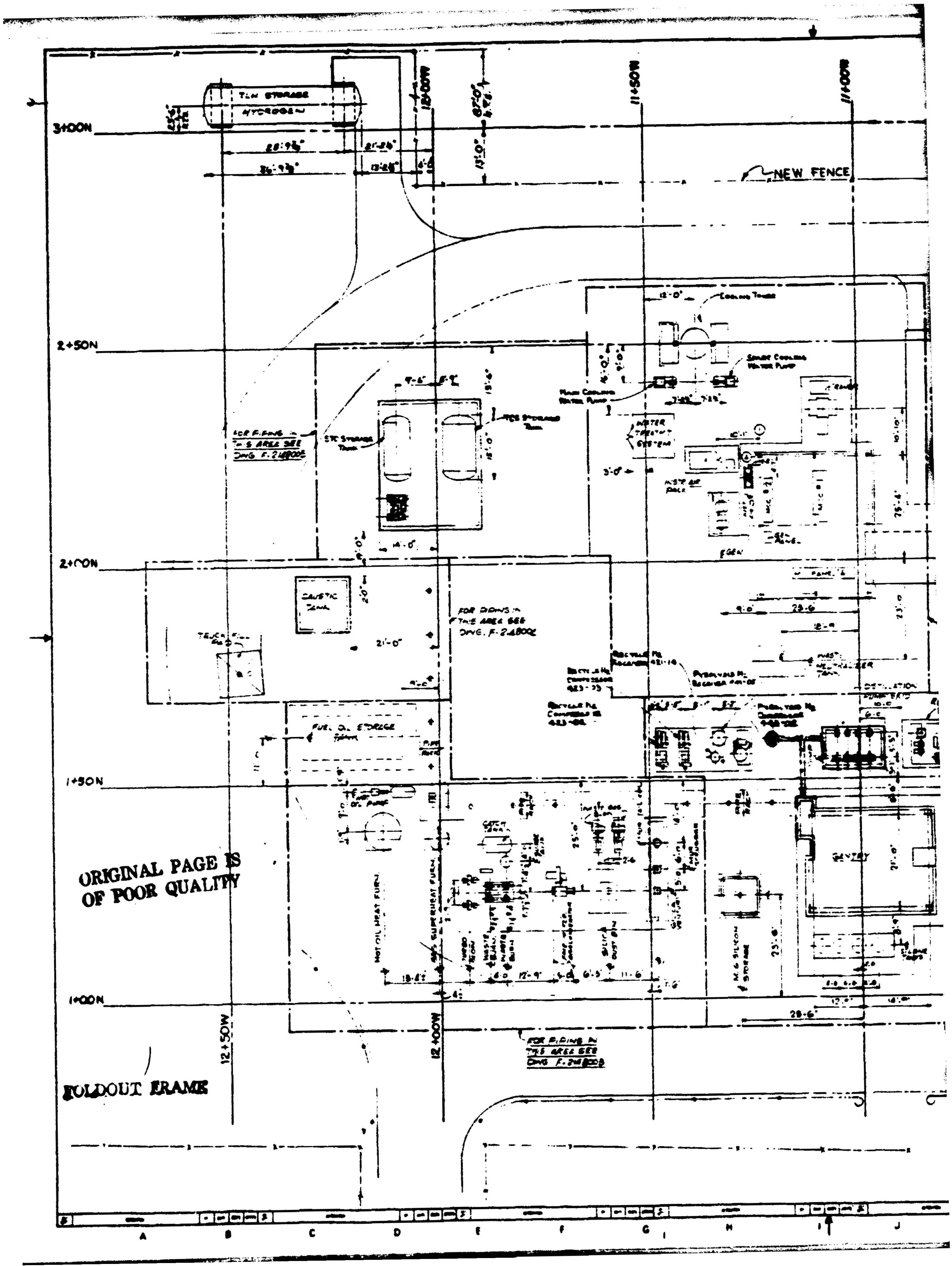


FIGURE 1 GANTRY SCALE MODEL



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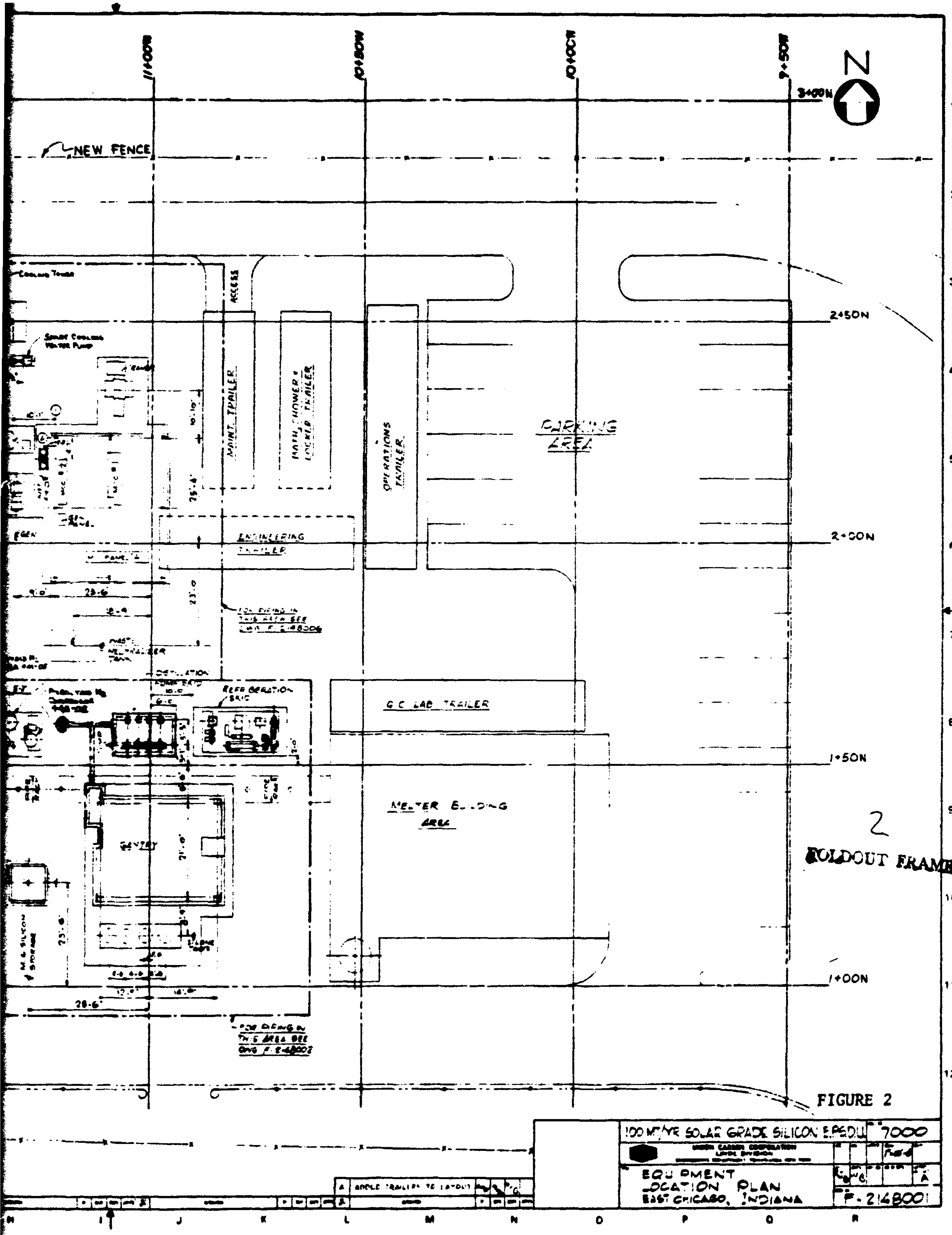

FIGURE 2

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1.1.3 Equipment Design, Specification, Procurement

The equipment related effort includes development of the control system, preparation of the piping and instrumentation diagram, preparation of wiring schematics and control panel drawings, and the design of equipment. The specification activity includes definition of specific requirements for each item of equipment, preparation of bid packages, evaluation of vendor quotation, and preparation of final specifications and drawings. Procurement includes the issuance of procurement packages to selected vendors and obtaining comprehensive design information necessary for preparing installation drawings.

The design and procurement of each item of equipment is accomplished through the combined efforts of process engineers, equipment engineers, and purchasing agents. These efforts produce a series of documents that evolve, ultimately, into a complete, definitive procurement package. The individual documents and their respective uses are as follows:

- Functional Specifications: This specification is developed by the process engineer based on process requirements reflected in the process flow diagram, heat/mass balance, and process control scheme. It defines the duty that this item of equipment must perform for the overall process system to operate.

- Engineering Specifications: Using the Functional Specification as a basis, the equipment engineer determines the specific type of equipment necessary to satisfy the process requirements. This translation of process requirements into hardware-specific information is delineated in the engineering specification.

- Request for Quotation (RFQ): The Request for Quotation (Form PUR 201), prepared by the equipment engineer, summarizes the equipment requirements, identifies vendors to be contacted, and defines the bidding instructions. This form, plus the engineering specification, constitutes the RFQ package submitted to the purchasing department for transmittal to potential

vendors. Vendor quotations are reviewed by the equipment engineer and, based on a technical, cost and schedule evaluation, a vendor is selected.

- Request for Requisition (RFR): The Request for Requisition, prepared by the equipment engineer, consists of the RFQ package plus the Bid Evaluation Report that identifies the selected vendor and the specific equipment model to be purchased. The RFR is submitted to purchasing and serves as the technical basis for the purchase order.

- Purchase Order (PO): The Purchase Order (Form L334-31DO), prepared by the purchasing agent, definitizes the terms and conditions, delivery requirements, and billing instructions applicable to the particular equipment and vendor. This form supplements the technical information contained in the RFR to provide a complete procurement package. When the internal review cycle is completed and the appropriate approvals have been obtained, the validated Purchase Order is issued to the vendor.

- Procurement Status Report (PSR): After the order is placed technical performance is monitored by engineering personnel and contractual performance is monitored by purchasing personnel. Status is reflected in the Procurement Status Report. (The PSR for December, presented in Appendix A, reflects the current procurement status for all EPSDU equipment).

These six documents serve as milestones for measuring performance of the procurement cycle for each item of equipment.

1.1.3.1 Process Control

The controls systems engineering effort includes all activities associated with developing the P&I diagram, designing process control loops and control panels, specifying valves and instrumentation, and preparing control wiring and pneumatic tubing diagrams.

A marked-up version of the P&ID was issued which collects all comments and corrections to previous versions. The comments and corrections are the outcome of an intensive series of review meetings during December.

The schematic wiring diagram was updated and re-issued to assist the electrical design group with their design and drafting effort.

The main control panel design was completed and issued to vendors for quotations. Figure 3 represents the design of the operating/front face of the panel that will be located in the switchgear-control room.

Programming of the Modicon controller was started, along with an "audit" of all process control system documentation to ensure that documents are consistent and all required instrumentation components are on order.

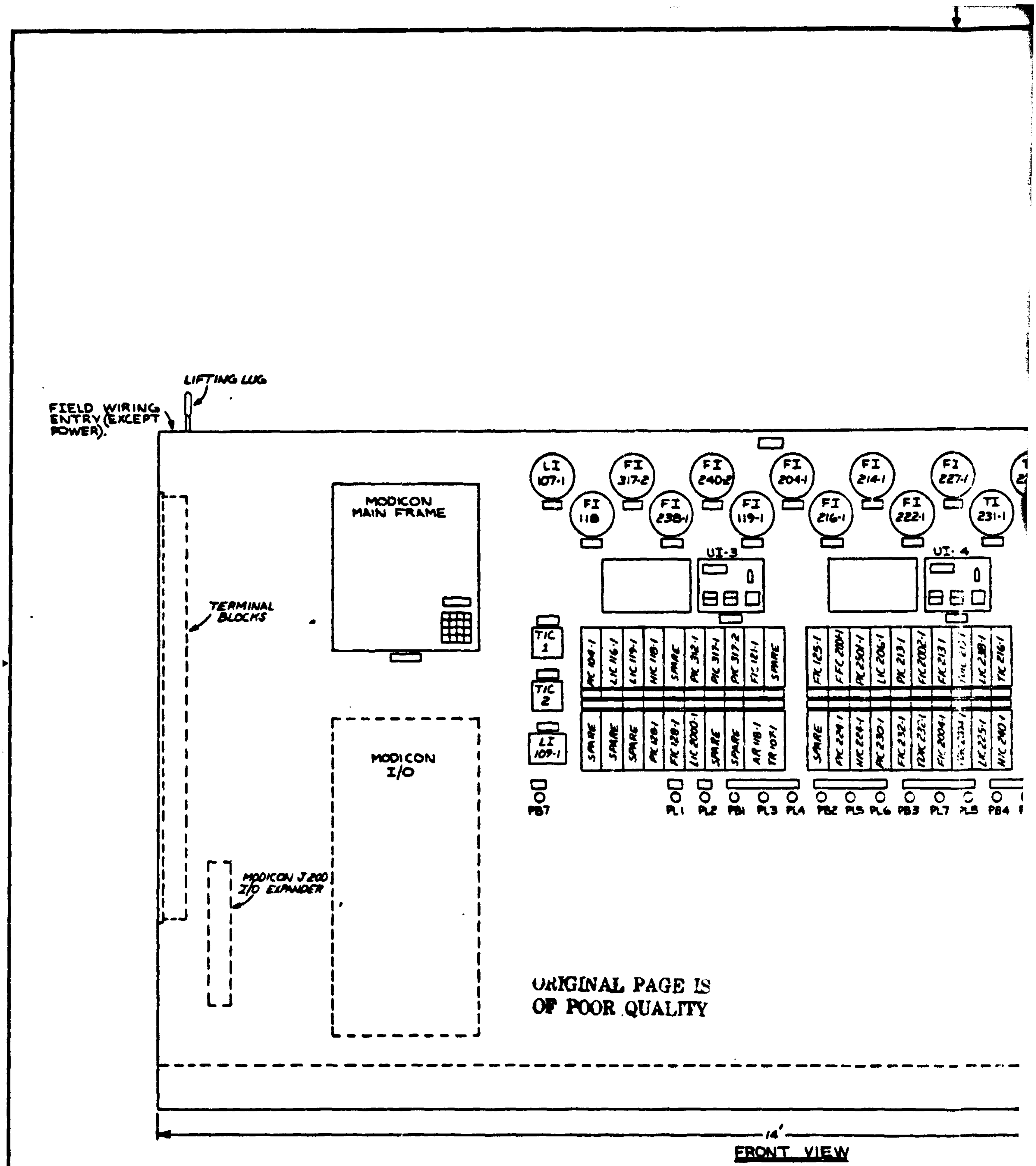
Four local input/output panels have been designed and issued for quotations.

1.1.3.2 Electric Power System

This effort included all electrical engineering activity required to develop the power control and distribution systems, prepare the definitive electrical one-line diagram, and specify electrical equipment. All activities covered by this WBS item were completed in the second quarter.

1.1.3.3 Process Equipment - UCC Designed

This effort includes the in-house activity necessary to prepare a complete design for specialized equipment such as the redistribution reactors and distillation columns. This effort was completed during the second quarter.



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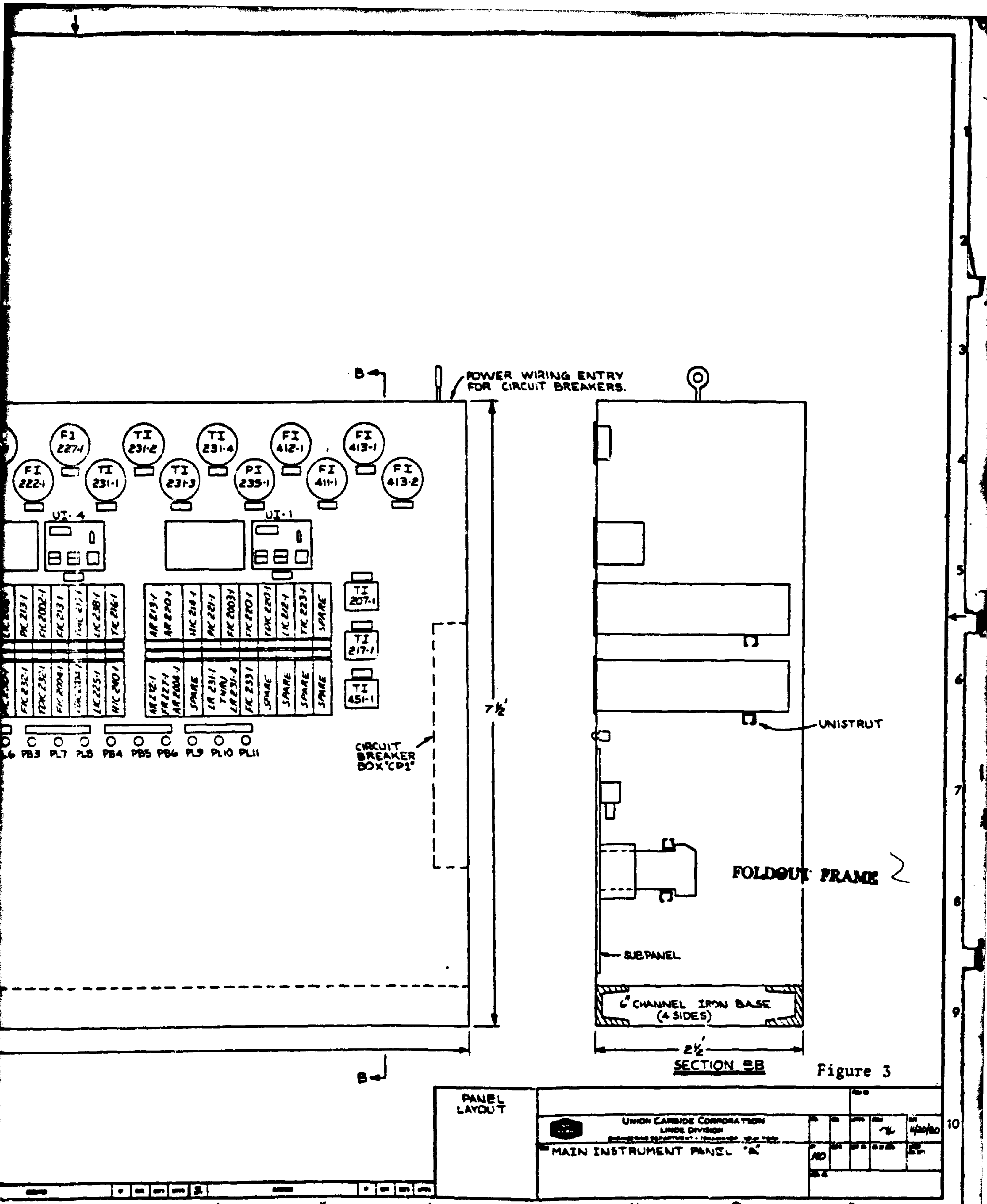


B
POWER WIRING ENTRY FOR CIRCUIT BREAKERS.
FI 227-1
TI 231-2
TI 231-4
FI 412-1
FI 413-1
FI 222-1
TI 231-1
TI 231-3
PI 235-1
FI 411-1
FI 413-2
UI-4
UI-1
TI 207-1
TI 217-1
TI 451-1
PB3 PL7 PL8 PB4 PB5 PB6 PL9 PL10 PL11
CIRCUIT BREAKER BOX "CP1"
7½'
UNISTRUT
SUBPANEL
6" CHANNEL IRON BASE (4 SIDES)
2½'
SECTION BB
FOLDOUT FRAME
PANEL LAYOUT
UNION CARBIDE CORPORATION
LINDE DIVISION
MAIN INSTRUMENT PANEL "A"
H I J K L M N O P
1 2 3 4 5 6 7 8 9 10

Figure 3

1.1.3.4 Process Equipment - Vendor Designed

This activity includes the engineering effort associated with specifying and selecting process equipment such as compressors, pumps, and tanks that will be designed and fabricated by commercial suppliers.

Request for Requisition was issued for the Caustic tank immersion heater and the Silica dust filter bags.

The purchase order for the waste burner was issued and a meeting was held with the vendor to determine the schedule of drawings and information flow. To insure that the control/safety wiring information could flow well, a meeting was held with the vendor's engineering representative to establish a technical link.

The silica agglomerator drawings and specification were completed and the RFQ was issued in December 1980.

1.1.3.5 Auxiliary Equipment

This includes the engineering activities associated with specifying and selecting auxiliary equipment such as heating and refrigeration systems, instrument air unit, and cooling tower that will be designed and fabricated by commercial suppliers. All effort covered by this WBS activity was completed in the second quarter 1980.

1.1.3.6 Support Equipment

This effort includes the engineering activities associated with specifying and selecting commercially supplied equipment such as trailers, materials-handling systems, and specialty items. All effort covered by this WBS activity was completed in the second quarter 1980.

1.1.3.7 Other Equipment, Specialties

Some new specialty items were added to the list. The addition to the specialties list (now believed to be a complete list) is:

- Chlorosilane Pump Seal Systems: All major parts received at Tonawanda.
- Hydrogenation Reactor Ram Valve: Ordered with anticipated delivery in mid-January for fitting to a seal pressure booster and control instrumentation.
- Sintered Metal Process Filters: Received and will be shipped to vendor or field after the installation design is firm.
- Sample conditioners and tube furnace.
- Silica Agglomerator Cleaner: New item to be designed and procured.
- Silica Venturi Gas Inlet Spool Piece: New item to be sized and procured.
- Waste Burner Atomizers and Mounting Plates: New item to be designed and procured.

The TFE spool piece for the waste burner piping was specified and the process control schematic for the hydrogenation reactor ram valve was prepared. Preparations are underway to assemble specialty items in Tonawanda in next quarter.

1.1.3.8 Data Collection System

This work item covers all effort to tailor and specify a computerized data-collection system for EPSDU.

The Nova IV minicomputer, 2 CRTS, a line printer, twenty 5-megabyte disk packs, and 10 double-density floppy disks (diskettes) have been received at Tonawanda. Installation of the minicomputer and peripherals has been completed. The 5-megabyte disk packs and the floppy disks have been initialized and formatted on the Nova. This procedure establishes the record length, record density, and labelling to permit random access of data stored in the disks and diskettes. The software provided by Data General and standard software developed by UCC have been loaded into the permanent memory. This software includes utility programs and library files.

Installed in the same area as the peripherals is a Hewlett-Packard gas chromatograph electronic controller and remote I/O station. The Hewlett-Packard interface components will be installed and tested to ensure proper interfacing between the controller and the Data General minicomputer.

The process variable list was completed and stored on a disk pack in the minicomputer. Programs which allow the listing, sorting, and the alteration of the list were also transferred onto this disk pack. This table is a standard UCC method for setting up a data storage bank on a minicomputer. The process variable list includes an internal identification number, an external identification number, a description, engineering units, signal type, signal location (Panel Number), an instrument tag, and the variable class. A sample sheet is attached (Table II). The two identification numbers are the minicomputer's primary means of searching for a specific variable. The description is the users primary means of searching for a specific variable. The units are the storage locations of the process variable value. Signal type is the type of signal which is sent from the instrumentation:

PN = pneumatic (psig)
T/C = thermocouple (mvolts)
E = electronic (mamps)

The panel locations have not been assigned at this time. The instrument tag is the identification number of the instrument from which the signal originates. The variable class identifies the type of scanivalve system which the signal is collected from:

TABLE II

SILICON EPSDU PROCESS VARIABLE LIST

11/11/80

ID		ED	DESCRIPTION	UNITS	SIG	PANEL	INST.TAG	CLASS	COMMENT
PV	1	ABT071	MAKE-UP H2 TO HYD REA TEMP	DEG F	E	-	TE107-1	4	S RTD
PV	2	ABT072	HYDROGENATION REA TEMP #1	DEG F	E	-	TE107-2	4	S RTD
PV	3	CDP172	MAKE-UP H2 TO HYD REA PRES	PSIG	PN	-	PT317-2	3	
PV	4	ABT231	SUPHEA STC TO HYD REA TEMP	DEG F	E	-	TE123-1	4	S RTD
PV	5	ABF281	STC FLOW FROM STC STORAGE	ACFM	PN	-	FIT128-1	3	WALL & TIER
PV	6	ABT221	SUPHEAT H2 TO HYD REA TEMP	DEG F	E	-	TE122-1	4	S RTD
PV	7	ABF211	COMPRSD H2 FLOW TO SH FURN	ACFM	PN	-	FT121-1	3	WALL & TIER
PV	8	ABT073	HYDROGENATION REA TEMP #2	DEG F	E	-	TE107-3	4	S RTD
PV	9	ABT074	HYDROGENATION REA TEMP #3	DEG F	PN	-	TT107-1	3	
PV	10	ABL071	HYDROGENATION REA LEVEL	PSIG	PN	-	LT107-1	3	D/P CELL
PV	11	ABD072	HYDROGENATION DELTA P	PSIG	PN	-	PDT107-2	3	D/P CELL
PV	12	ABP071	HYDROGENATION INLET PRESS	PSIG	PN	-	PT107-1	3	
PV	13	ABT081	HYDROGENATION OUTLET TEMP	DEG F	E	-	TE108-1	4	S RTD
PV	14	ACT082	WASTE SETTLER TANK TEMP	DEG F	T/C	-	TE108-2	5	TYPE T
PV	15	ACT111	RECYCLE TO QC VENTURI TEMP	DEG F	T/C	-	TE111-1	5	TYPE T
PV	16	ACF171	QUENCH CONDENSER WASH FLOW	ACFM	PN	-	FIT117-1	3	WALL & TIER
PV	17	ACD132	QUENCH CONDENSER FEED DP	PSIG	PN	-	FT113-2	3	D/P CELL
PV	18	ACT141	QUENCH RECEIVER TEMP	DEG F	T/C	-	TE114-1	5	TYPE T
PV	19	ACF191	CRUDE TCS FLOW TO STORAGE	ACFM	PN	-	FIT119-1	3	WALL & TIER
PV	20	ACY161	VALVE POS ON RECYLCE TO QC	% OPEN	PN	-	-	3	NOT SPEC
PV	21	ACP181	H2 PRESS IN RECYCLE RECEIV	PSIG	PN	-	PT118-1	3	
PV	22	ACT181	H2 TEMP IN RECYCLE RECEIV	DEG F	T/C	-	TE118-1	5	TYPE T
PV	23	ACL251	TCS STORAGE TANK LEVEL	FEET	-	-	LT125-1	0	DREX/DIRECT
PV	24	ABT241	SAT. STC FROM VAPORIZOR T	DEG F	T/C	-	TE124-1	5	TYPE T
PV	25	ABT075	HYDROG REA STC FEED TEMP	DEG F	E	-	TE107-5	4	S RTD
PV	26	AAL001	LEVEL OF M.G. SILICON BIN	FEET	-	-	LI100-1	0	DIRECT
PV	27	BAF251	STRIPPER COLUMN FEED FLOW	ACFM	PN	-	FIT125-1	3	WALL & TIER
PV	28	BAT257	STRIPPER COLUMN FEED TEMP	DEG F	T/C	-	TE125-7	5	TYPE T
PV	29	BAT251	STRIPPER COLUMN TEMP #1	DEG F	T/C	-	TE125-1	5	TYPE T
PV	30	BAT252	STRIPPER COLUMN TEMP #2	DEG F	T/C	-	TE125-2	5	TYPE T
PV	31	BAT253	STRIPPER COLUMN TEMP #3	DEG F	T/C	-	TE125-3	5	TYPE T
PV	32	BAT254	STRIPPER COLUMN TEMP #4	DEG F	T/C	-	TE125-4	5	TYPE T
PV	33	BAT255	STRIPPER COLUMN TEMP #5	DEG F	T/C	-	TE125-5	5	TYPE T
PV	34	BAT256	STRIPPER COLUMN TEMP #6	DEG F	T/C	-	TE125-6	5	TYPE T
PV	35	BAT031	STRIPPER OVHD TEMP	DEG F	T/C	-	TE203-1	5	TYPE T
PV	36	BAT041	STRIPPER CONDENS LIQD TEMP	DEG F	T/C	-	TE204-1	5	TYPE T
PV	37	BAT501	STRIPPER CONDENS VAPR TEMP	DEG F	T/C	-	TE2501-1	5	TYPE T
PV	39	BAF501	STRIPPER CONDENS VAPR FLOW	ACFM	PN	-	FT2501-1	3	WALL & TIER
PV	40	BAD041	STRIPPER REFLUX DP	PSIG	PN	-	FT204-1	3	ORIFICE

0 = not through the scanivalve system
3 = pneumatic scanivalve
4 = electronic scanivalve
5 = thermocouple scanivalve

The process variable list (the minicomputer listing of data points) and the newest P&I were reviewed to ensure that all data needed to calculate design parameters (conversion rates, efficiencies, mass balances, etc.) was included. Both documents required alteration.

The process I/O system was specified. This system is the interface between the process equipment (thermocouples, flow meters, level indicators) and the minicomputer. Most of the signals (184 signals) will be routed through a scanivalve system which has four modules — one thermocouple, one electronic, and two pneumatic.

Signals from these modules travel through four channels on an analog-to-digital (A/D) card, the resulting proportional digital signal enters the computer. There are approximately 20 analog signals which do not go through the scanivalve system but are directly associated with a dedicated channel on an A/D card. There is an economic balance between the cost of the channels and the cost of an additional scanivalve module; the system described above has been chosen as the most efficient and reliable.

The digital signals which enter the computer are converted into values in engineering units (DEG F, psig, ACFM, etc.); these values are then stored on a disk pack. The tables and information required for these conversions were transmitted to the Computer group for loading into the minicomputer memory.

A system for data reduction operation at Tonawanda has been identified. The full disk packs of data obtained at EPSDU will be sent to Tonawanda. The data will be transferred to a magnetic tape and will then be read into the UCC main-frame computer network. A combination of sorting and statistical routines will be used to reduce the data to a useable form. The data will then be entered into several project-specific engineering programs which will produce operations information and design parameters.

The final process variable list was issued to the Computer Applications Group for their use. The process groups work will now shift to preparing some of the support/report generation/data reduction routines while the Computer Applications Group prepares the data acquisition/storage routines. The first of these support routines will be a minimal physical properties package to allow data to be expressed in meaningful units and area mass balances to be tabulated. An earlier engineering memorandum, EM-6206, by T. E. Diegelman, "Physical and Critical Properties of Silane and the Chlorosilanes" will be used as a data source.

Initial problems of structuring data collection files, calibration sampling frequency have been resolved.

..1.4 Installation Design, Specification, Subcontract

This design effort includes development of separate installation drawing packages for the site, civil, mechanical, and electrical specialties based on the engineering design effort and vendor-supplied information. Specification activity includes definition of specific requirements for performing all installation functions. Subcontracting includes the preparation of bid packages, evaluation of quotes, sub-contractor selection and contract negotiation.

1.1.4.3 Civil Drawings

The preparation of drawings for civil/structural package is complete.

1.1.4.4 Mechanical Design, Specification and Materials

Design work on specification of piping components and materials, including manual valves, check valves, safety relief valves, and piping and equipment insulation was completed in November. Determinitation of the size and location of some thirty safety relief valves was resolved.

The high cost of "packless" shut-off valves which are required for silane streams will necessitate additional vendor selection and a review of

the minimum number of valves for practical operation/cost effectiveness. Suppliers of these special valves quoted prices from five to ten times higher than "standard" valves.

Cleaning and testing procedures for all piping components exposed to chlorosilanes (moderate cleaning required) and silane (very thorough cleaning required) were determined.

Other installation specifications which require definition include pressure testing procedures, welding procedures, painting/surface protection, and location of liquid drains. These activities are on going.

RFR's were issued for various valves (25 plug, 145 globe, 253 gate and 67 check).

1.1.4.5 Mechanical Drawings

During this quarter the initial draft of the mechanical drawing package was completed and received the scrutiny of all engineering disciplines including the design safety review team. Recommended changes to the process and control piping impacted the cost and schedule to complete the mechanical design activities.

The EPSDU design review meetings were completed in December. All comments from the Process Design, Control Systems Design, Equipment Design and Mechanical Design Groups were collected on a master set of piping and equipment drawings. A considerable number of major and minor changes to the mechanical design package were identified which will require approximately three additional months of design and drafting work.

Aspects of the design which will be changed include:

- Locations and configurations of safety valves.
- Simplified waste header and pressure relief vent header.
- Elimination of redundant shut-off valves.
- Elimination of infrequently used piping components such as by-pass loops.

- Addition of low-point drain valves.
- Addition of flow check valves.
- Relocation of therminol piping at reboilers.
- Relocation of piping at STC/TCS tanks.
- Elimination of non-essential instrumentation.
- Elimination or relocation of purge valves.
- Relocation of interconnecting process piping.
- Elimination of heat tracing on water and sample lines.

All of these changes are necessary to improve operability and maintenance and to reduce piping installation costs.

1.1.4.6 Electrical Design, Specifications, Materials

An engineering design team meeting was held in November to review progress-to-date on all electrical design activities.

The electrical one-line diagram was updated to reflect vendor drawing information and issued. The electrical design effort is continuing and will be updated as necessary to be compatible with any changes to the final mechanical design.

1.1.4.7 Electrical Drawings

Locating and labeling of all electrical process control instruments and devices on drawings proved to be an involved task due to the extensive process control/data collection system designed and specified for EPSDU. The location of each locally-mounted instrument was identified; cable-tray and conduit locations were identified and detailed drawings are being developed.

The preliminary version of the electrical design package has been completed and placed on hold pending changes to the mechanical design package.

1.1.5 Cost Estimating

Cost engineers support the design effort by providing cost estimates and controls for procuring equipment and installing the EPSDU.

1.1.5.1 Facility Cost Estimate

Complete.

1.2 EQUIPMENT FABRICATION/DELIVERY

This report item includes all in-house and outside activity associated with fabrication, delivery, and vendor coordination for all items of equipment.

1.2.1 Process Control & Data Collection System

This task includes the effort required for the fabrication, delivery and acceptance of field instruments and controls, panel and panel instruments, data collection system, and other process control equipment.

1.2.1.1 Field Instruments and Controls

The following validated Purchase Orders were issued this quarter:

825-50165 Flow Indicators (47 items)
825-50177 Orifice Plate
825-50181 Flow Indicators (8 items)
825-50182 Valve Manifolds (18 items)
825-50183 Pressure Gauges (10 items)
825-50197 Load Cells (4 items)
825-50202 PH Controller
825-50205 Gas Regulators (2 items)
825-50178 Pressure Transmitter

825-50212 Temperature Controllers
825-50214 Flow Tube
825-50179 Pressure Switches (7 items)
825-50200 Level Switches (2 items)
825-50199 Flow Elements (8 items)
825-50215 Sludge Indicator
825-50184 Pressure Switches (22 items)
825-50185 Transducers (8 items)
825-50201 N_2/H_2 Analyzer
825-50213 Regulator

1.2.1.2 Panels and Panel Instruments

The following validated Purchase Orders were issued this quarter:

825-50166 Panel Instruments (48 items)
825-50167 Pressure Gages (5 items)
825-50169 Shutdown Monitors (4 items)
825-50174 Solenoid Valve
825-50170 Pressure Transducers (8 items)
825-50172 Safety Barriers (29 items)
825-50165 Pressure Switches (27 items)
825-50173 Isolator Transmitter
825-50176 Temperature Switches (2 items)
825-40175 Milliamp Meter

1.2.1.4 Other Process Control Equipment

The following Purchase Orders were issued:

825-50190 8" and 10" Automatic Valves (4 items)
825-50193 1/4" and 1/2" Automatic Valves (13 items)
825-50189 Automatic Valves (2 items)

1.2.3 Process Equipment - Linde Designed

This task includes the effort required for the fabrication, delivery, and acceptance of the free-space reactor with hopper, hydrogenation and redistribution reactors, distillation columns, and other process equipment.

1.2.3.2 Reactor

Estimated shipment date is 16 January 1981.

1.2.3.3 Distillation Columns

Construction of the four columns is in progress — estimated shipment date is 15 February 1981.

1.2.4 Process Equipment - Vendor Designed

This task includes the effort required for the fabrication, delivery, and acceptance of compressors, pumps, heat exchangers, tanks, solids handling equipment, waste treatment equipment, and other process equipment.

1.2.4.1 Compressors

Purchase orders were placed in the second quarter for delivery in January 1981.

1.2.4.2 Pumps

The purchased pumps are to be shipped in January 1981.

1.2.4.3 Heat Exchangers

The heat exchanger estimated shipping date is January 1981.

1.2.4.4 Process Equipment/Tanks

The following Purchase Orders were issued:

825-50160	4500-gallon caustic storage tank
825-50055	200-gallon therminol expansion tank
825-50162	350-gallon fuel oil tank
825-50035	(reissued) process tankage (9 items)
825-50040	(reissued) silane tanks (4 items) redistribution reactors (2 items)

1.2.4.5 Process Equipment/Solids Handling

P. O. 825-50164 1 inch M.G. silicon ram valve

1.2.5.6 Other Auxiliary Equipment

The following Purchase Orders were issued:

825-50155	In-line filters, flow restrictors (79 items)
825-50156	Solenoid valves (9 items)
825-50161	Caustic Immersion Heater

1.2.6 Support Equipment

This task includes trailers outfitted for quality control, maintenance, and offices.

1.2.6.1 Q.C. Trailer

Purchase order 825-50096 was issued on November 5/80 for shipment in February 1981.

1.2.8 Procurement Support

This task includes the Procurement Department effort necessary to initiate, monitor and control the purchase of equipment.

1.2.8.1 Purchasing, Expediting

Monthly procurement status reports were issued. To date, approximately 116 validated purchase orders have been issued for equipment. Equipment is being delivered to the EPSDU site.

1.2.8.2 Equipment Design, Inspection

Drawings submitted by vendors were reviewed by appropriate engineering team members for approval, alteration, and/or correction. The final series of approved vendor drawings was completed in December.

Shop visits to vendors who have begun to fabricate custom process equipment such as tanks, bins, reactors, and heat exchangers began in November. This equipment will be inspected to ensure conformance with our detailed specifications.

1.2.8.3 Vendor Print Control

Vendor drawings discussed above were received, routed through the various engineering disciplines, and returned to vendors. The monthly log sheet was issued.

1.3 INSTALLATION AND CHECKOUT

This report item includes all effort associated with award of subcontracts, providing instructions to the on-going subcontract activities, monitoring subcontractor performanee, and checkout activities to ensure proper installation.

1.3.1 Installation Subcontracts

This task includes the effort for all installation activities assigned to subcontractors and includes labor, materials, and day-to-day job supervision.

1.3.1.1 Site

Complete.

1.3.1.2 Civil, Underground, and

1.3.1.3 Structural

All equipment and building foundations are complete and all steel has been erected for the Gantry Structure. The contractor has completed the installation of the 12.5 KV power line, the 3-inch natural gas line, lighting poles and connecting conduit.

The 2-inch nitrogen gas supply and the 4-inch sewer line connecting the EPSDU in the specialty gas plant was also installed. All underground electrical work is complete. The piping subcontractor completed all underground piping with the exception of four fire hydrants which will be installed in January.

The only major activity remaining is installation of the pre-engineered switch gear/control room building and the melter building. Both structures will be completed in January and will be used to store equipment as it is received.

1.3.1.4 Mechanical and Piping

Major equipment items scheduled to be delivered starting in January will be stored in the Melter Building (unheated), in an adjacent Linde-owned warehouse (heated), or set in place on foundations. A purchase order was issued to Linde Gas Products/East Chicago to have their in-house maintenance contractor, Calumet Industries, perform all work associated with off-loading, rigging, storing, and/or setting equipment.

1.3.2 Installation Materials

The activities associated with specifying and requisitioning these special installation items fall under work item 1.1.4.4 (Mechanical Design) if the items pertain to process piping and under work item 1.1.4.6 (Electrical Design) if the items pertain to electrical wiring.

Work items under 1.3.2 is used to identify the purchase orders and the associated cost of these items. Items purchased under this category generally represent long-lead, large quantity, and/or special items better purchased by UCC than the contractor to ensure the timely delivery and proper type, and to avoid subcontractors mark-ups. We no longer plan purchases under 1.3.2.1 (Structural) and this will be an inactive work item. All structural items will be supplied by installation subcontractors.

We plan to purchase manual on-off valves, check valves, safety valves, heat-traced SS sample tubing, 347 SS piping, and other specialty items under 1.3.2.2 (Mechanical Piping).

Evaluation of long-lead or specialty items to be purchased under 1.3.2.3 (Electrical) and 1.3.2.4 (Other) took place during the November review meetings.

1.3.2.2 Mechanical, Piping

Validated purchase orders were issued as follows:

825-50188	Plug Valves (25 items)
825-50194	Check Valves (71 items)
825-50207	MG Silicon Transfer Hose

Purchase orders for additional piping materials and components will be issued as the drawing work is completed to ensure that the proper type and number of items are obtained.

1.3.3 Installation Support

This task includes the in-house support associated with supervising, directing, and monitoring the installation effort.

1.3.3.1 Field Supervision

The field office was fully operational with installation of electric power and telephones in mid-October. The field construction manager, David L. Schmiege, arrived on-site in late October.

The construction manager will coordinate and be responsible for all field activities. Weekly meetings are being held with the contractor to ensure effective communication. Written progress reports are also issued weekly which note significant highlights.

JPL and UCC Program Staff visited the EPSDU job site in early December.

The field staff will be two full-time people effective January, 1981 to correspond to the reduced level of construction activity during the first several months of 1981.

1.3.3.5 Procurement

This activity includes the effort required to award subcontracts, monitor and evaluate subcontractor performance, and determine final compliance with subcontract provisions.

1.6 PROCESS SUPPORT R & D

The supporting R & D Program is separate from the mainstream design effort and includes all activities associated with analytical and experimental development of the free-space reactor, melting/consolidation system, fluid-bed reactor system, and quality control techniques and procedures. Information generated in this program will be used for the EPSDU effort and the commercial facility economic analysis.

1.6.1 Free-Space Reactor Development

This development task includes all experiments and analysis necessary to verify design data for the free-space pyrolysis reactor and to develop a new reactor model.

1.6.1.1 PDU Experiments

Construction and operation of the Tonawanda free-space reactor PDU had two explicit objectives: to demonstrate that a free-space reactor can operate reliably for extended periods, and to determine the maximum feasible throughput of the 8-inch reactor used in the PDU. Achieving these objectives would confirm the viability of the free-space reactor concept for EPSDU while providing a rational basis for scale up of the actual EPSDU reactor. In addition to these explicit objectives, it was assumed that the purity performance of the Parma free-space pyrolysis powder would carry-over to the Tonawanda PDU.

Purity & Operability Tests

Planned modifications to the free-space reactor PDU were completed and the unit was returned to operation in October. Modifications included installation of a new vacuum line to the hopper. The vacuum pump has been relocated to an outside shed and a 30-foot long 2-inch diameter copper line has been run to the PDU. This line is shared with the fluid-bed PDU that is currently being installed in the test cell. Several instrument lines were also rerouted to eliminate conflicts with fluid bed piping. Following a careful cleaning with deionized water, the free-space reactor was sealed on the 13th of October. Drying, purging, leak detection, and repair occupied the next 10 days, and the PDU was started up on the 23rd of October.

Three consecutive runs (Run 14, 15 and 16), accumulating a total of 11 hours were made which marked the completion of a milestone. Run 17 (12 hours duration) was made in November. The test results are summarized below:

Run Number	14	15	16	17
Date of Test	Oct. 23/81	Oct. 28/81	Oct. 31/81	Nov. 10/81
Peak Reaction Zone Temperature °C	890	895	875	874
Silane Flow kg/hr	2.30	2.31	2.29	2.40
Duration hours	3.0	4.0	4.0	12.0
Powder Collected kg	4.5	6.7	8.13	19.7

In all cases silane conversion exceeded 99.99% (no silane could be detected in the effluent). In all three tests no operational problems were encountered. Inspection of the hopper and reactor with a borescope showed the liner to be intact. The new powder transfer system is operating efficiently, much better than the older system. Following shutdown and purging, the powder was transferred and stored under a dry nitrogen blanket to avoid air and moisture contact.

Following the conveying effort the PDU was opened for inspection. Some powder was "hung up" above the porous cone inside the hopper. Only the area around the gas "cannon" was free of buildup. The porous cone appeared to have been effective in that it had undercut the deposits above it. It seems clear that an increased cone angle and an extension of the porous surface over the entire lower cone will eliminate the formation of such deposits.

The powder remaining in the hopper was vacuumed into drums and weighed. A mass balance around the PDU for runs 14 through 17 was then calculated:

	SiH_4	Si Produced	Si Recovered	H_2 Produced	H_2 Measured
Runs 14-17	53.75 kg	47.00 kg	39.05 kg	6.75 kg	7.05 kg (104.4%)
"Post-Campaign"			7.81 kg		
Total			46.86 kg (99.7%)		

The silane consumption was determined by weighing the silane cylinders and the silicon and hydrogen produced were then calculated from this figure. The hydrogen flow was measured with a dry test meter. A systematic error in estimating purge flows is probable as the measured hydrogen flow exceeded the calculated flow by approximately the same amount for each run. The "post-campaign" silicon recovered is the silicon removed by vacuuming after the system was opened. The silicon balance is excellent and the hydrogen balance acceptable.

Analytical results for Runs 14 - 17 are summarized in Table III. The results of the purity analyses are comparable with those of previous runs. It has become very clear that standard analytical methods are not adequate for our purposes. Melting of the powder followed by pulling of a single crystal and determination of a resistivity profile appears to be the only procedure capable of determining the suitability of free-space reactor silicon for solar applications. The results of the BET tests are of interest. Although the two samples shown are not significantly different in particle size, they both differ dramatically from the powder produced in Run 3 which had a mass average particle size of 0.92 µm. The very large increase in temperature in the upper section of the reactor caused by modifications made to the PDU during Phase II operations, is undoubtably responsible for the dramatic decrease in particle size.

Examination of the quartz reactor liner used for Runs 14 - 17 revealed a small zone of opaque silicon deposit 0.6 m below the reactor head. There was some concern expressed by JPL, that these deposits could present a threat to the liner during extended operation. However, scanning electron microscope examination of the deposits revealed their maximum thickness to be less than 0.5 µm after 23 hours of operation. Extrapolating this growth rate to the 1-month life expected for liners at EPSDU give a thickness of 15 µm, which is over two orders of magnitude below the thickness that might present a problem.

TABLE III - FSR PDU ANALYTICAL SUMMARIES

RUN NO.	DATE	SiH_4 RATE (kg/hr)	PARTICLE SIZE [1]	PURITY (PPM) [2] Ni	Cr	Mn	Fe
14	10-23-80	2.30		0.9	0.20	0.08	9.0
15	10-28-80	2.33	0.34 µm	0.48	0.60	0.25	22
16	10-31-80	2.29	0.34 µm	1.4	0.15	0.10	11
17	11-10-80	2.37		1.3	1.0	0.15	13
POLYSILICON CONTROL				0.10	0.76	0.50	3.3

1 BASED ON BET SURFACE AREA. ASSUMES SPHERICAL PARTICLES.

2 ATOMIC ABSORBTION (Fe USING FLAME; Cr, Mn, Ni USING GRAPHITE FURNACE)

Final Test Run

The 12-hour duration run was completed on December 22/80. This run was made using the new scraper seal, and was very smooth. The seal will be inspected in the near future. Peak reaction zone temperature was 880°C, with a silane flow of 2.4 kg/hour, again silane conversion was better than 99.99%.

Other changes made to the PDU for Run 18 were the addition of an extra insulation collar above the main insulating blanket and the removal of 3 inches of insulation from the bottom of the reactor. The effect of these changes was to steepen the temperature gradient in the upper section of the reactor, flatten the temperature peaks in the midsection of the reactor, and start the sharp drop-off in temperature closer to the top of the reactor. This profile is somewhat superior to that obtained in earlier runs, see Figure 4.

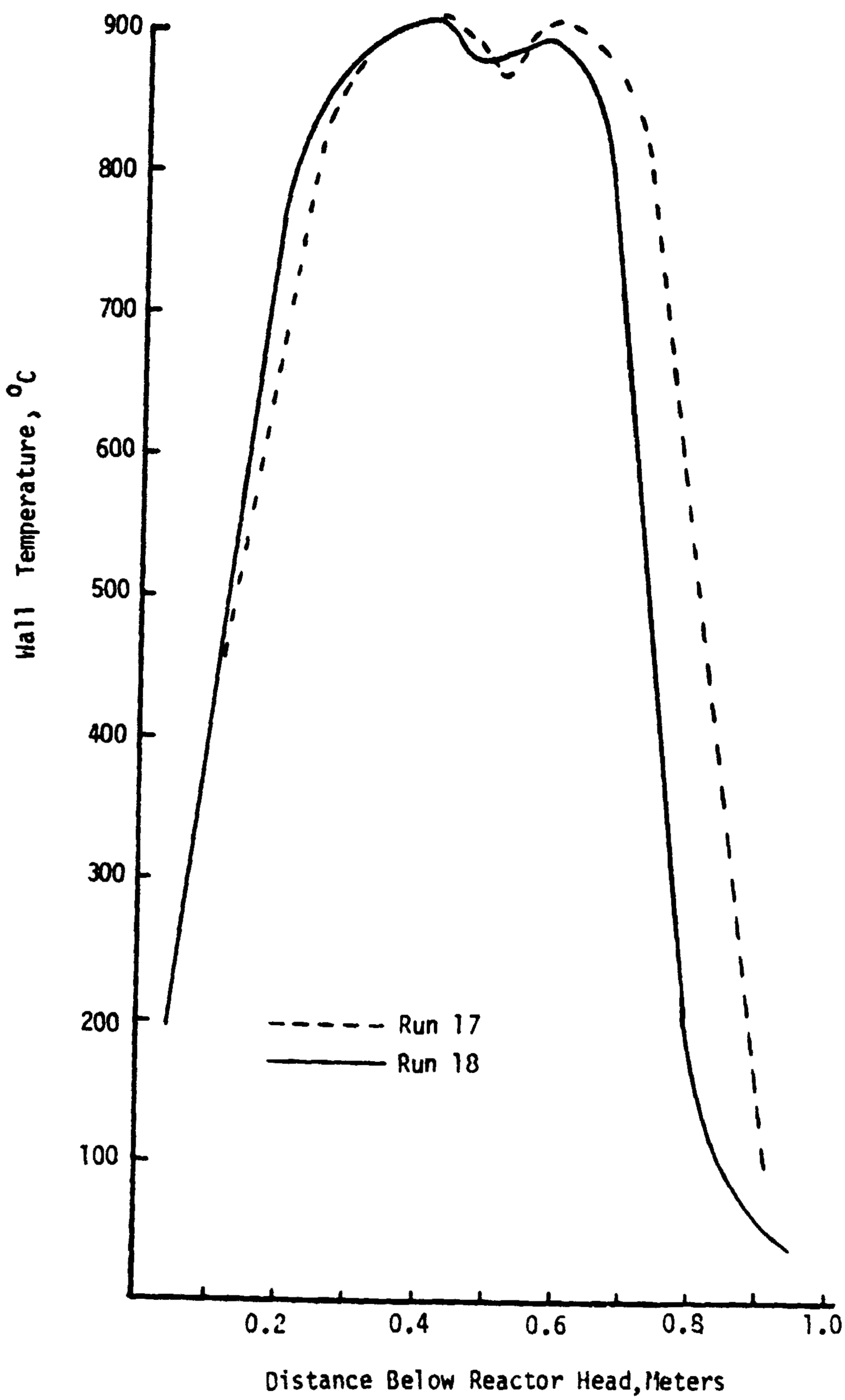


FIGURE 4 FSR-PDU Wall Temperature Profiles

Run 18 was the first test run to utilize the new scraper (see Figure 7). This scraper differs from that used in previous runs in that it uses an in-line double-ended air cylinder instead of a side-mounted single-ended cylinder, and separate elastomeric wipers and O-ring seals instead of simple packing rings. The new scraper should require less maintenance than the original unit. It also has a longer stroke — 0.75 m vs 0.45 m in the older scraper. Performance of the new scraper during Run 18 was smooth. The unit will be inspected for wear as soon as the unit is opened.

Run 18 was the last run called for in the FSR PDU program plan. Thus, all the runs planned for this phase were successfully completed and have demonstrated the viability of the system.

Following the depletion of available silane supplies early in January, the PDU will be shut down. Work has begun on the final report for the Phase II experiments and will be issued in the next quarter.

Powder Inventory and Shipping

Kayex fabricated a powder transport drum for powder shipments. This consisted of a highly modified stainless steel 55-gallon drum incorporating a screw auger, breaker bar, and porous fluidizing surfaces. The drum was used successfully by Kayex to transfer the powder to a melter crucible without exposure to the atmosphere.

Approximately 100 kg of silicon powder is required for shotting work by Kayex. Our current powder inventory (Dec 30/80) is 50 kg of good powder kept under nitrogen purge, and approximately 50 kg of powder with silicon chunks which have to be screened out. We, thus, have roughly 100 kg of powder for shotting work and no surplus in storage.

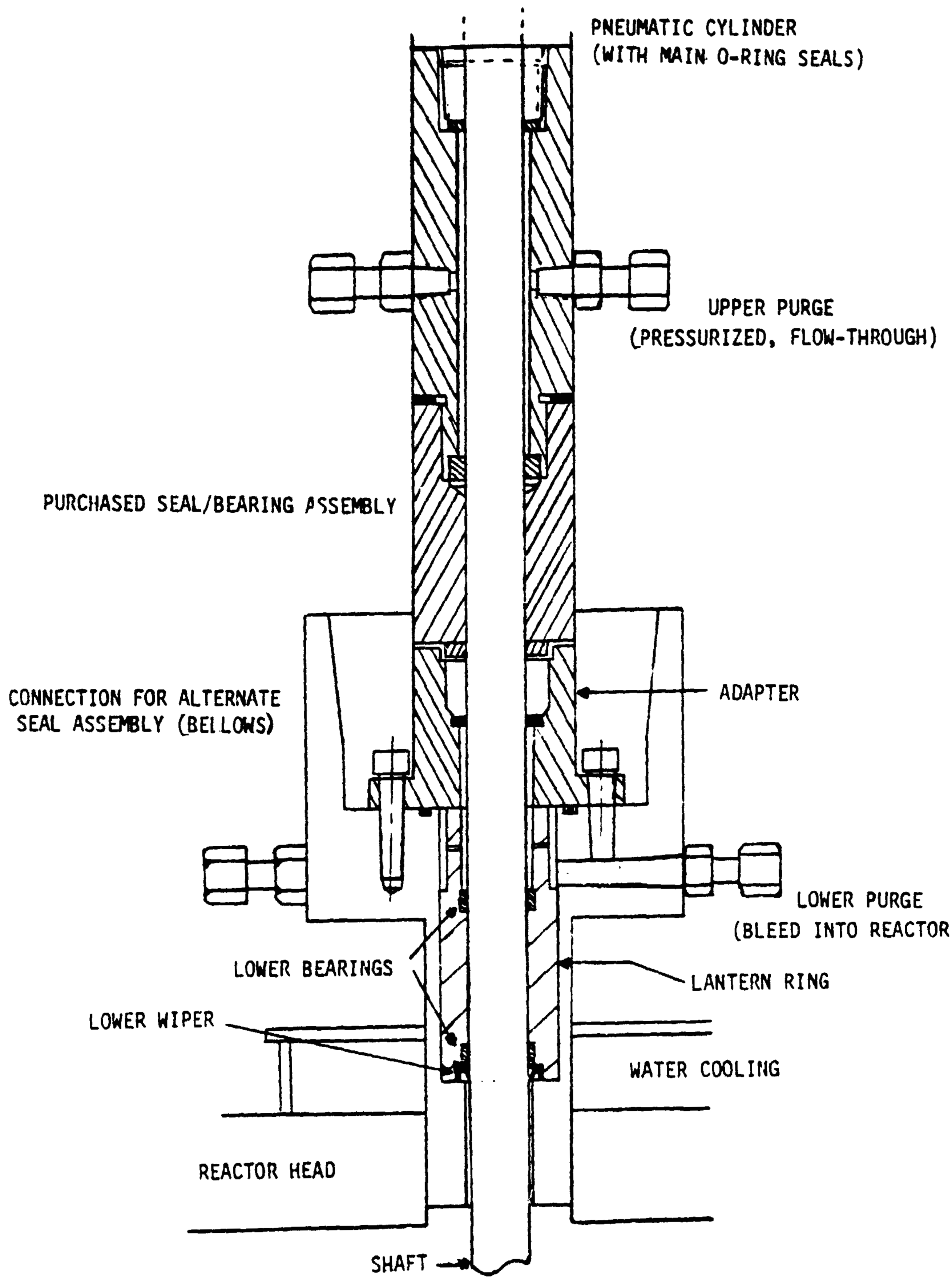


FIGURE 5 FSR New Scraper Seal

Reactor Scale-Up And Design

Computer simulations of the free-space reactor predict that an 18-inch diameter reactor is adequate for processing silane at the design throughput of EPSDU. This reactor will be heated by a multizone resistance heating system with automatic feedback temperature control. A computer model of black-body-radiation exchange between the resistance heaters and the reactor wall was developed to predict the temperatures and power inputs needed in the heating elements in the various zones to achieve the desired wall temperature profile. The temperature profile on the outside surface of the reactor wall is specified based on experimental results from the PDU, and the heat flux distribution on the inside surface of the reactor wall is determined via the computer program developed to study the fluid mechanics and heat transfer inside the reactor. The heat flux distribution on the outside surface of the reactor wall, which differs significantly from the inner distribution due to large axial heat losses out the reactor ends caused by the steep temperature gradients imposed near the ends, is determined by a finite element solution to the two-dimensional heat conduction problem in the reactor wall. Results indicate that about 35 kW of power must be supplied to the EPSDU reactor for 75 lb/hr silane throughput, 925^{o}C maximum wall temperature. About 20 kW of this power enters the reactor, heating the incoming gas, and the other 15 kW is lost out the reactor ends via axial conduction.

Results from the computer model conclusively show that reflective baffles between the independently controlled heating zones are an absolute necessity if the resistance heating system is to be capable of achieving the required temperature profile in the reactor wall. Without baffling, interaction between the heating zones becomes a serious problem. As the gap between the reactor wall and the heating elements becomes larger, interaction between the element becomes more and more significant, since the radiation view factors between adjacent zones increase. As the gap becomes larger, the required power input to the second zone increases until it exceeds the power rating of the heating elements. At the same time, the power input to the first zone becomes smaller until, eventually, heat actually flows out through the first zone. The temperatures in the second zone increase and those in the first zone decrease in the same manner as the gap is

increased. Baffling of the various zones to reduce interaction will have a similar effect to reducing the gap to a very small distance, and the heat flux distribution in the resistance elements should approach that in the outer reactor wall.

A simulation run of the updated model was made to determine whether the proposed EPSDU resistance heating system was capable of generating the wall temperature profile required in the EPSDU reactor. The reflectivity of the ceramic baffles and the ceramic backing of the heating elements was assumed to be 0.5, and the reactor wall was assumed to be black. A 2-inch gap between the heating elements and the reactor wall was specified.

Figure 6 shows the power inputs to the various heating zones, and Figure 7 shows the corresponding element temperatures needed to achieve the desired temperature profile. The maximum power input and element temperature was predicted in zone 3, with values of 6.8 kW and 994^{o}C respectively. Since each zone of heating elements has a power rating of 8 kW and a temperature capability of 1200^{o}C, the system is capable of achieving the desired profile. The negative power inputs to zones 1 and 12 indicate that heating elements are unnecessary in these regions provided these areas are properly insulated.

The first draft of our engineering memorandum describing the use of the theoretical model in designing the EPSDU reactor has been completed and is currently being reviewed. The report deals with the experimental verification of the free-space reactor model, the criterion used to scale up the reactor to EPSDU size, and the completed conceptual design of the EPSDU reactor and its associated multizone resistance heating system. The final version of the report will be issued in February.

Drafting work on the EPSDU free-space reactor and hopper was started. The reactor will utilize an 18-inch inside diameter quartz liner contained in an Incoloy 800 H reactor shell. The reactor head will contain a spring plate to provide a dust-seal to compensate for the approximately 1.25-inch differential expansion between the liner and reactor shell. Vendor contacts have been made to obtain the appropriate resistance-heating furnace.

The functional design of the EPSDU pyrolysis reactor and silicon hopper will be completed in February. Preliminary design drawings of the two vessels, and details about vendor-supplied components such as quartz liners, scraper-actuator, etc. will then be available.

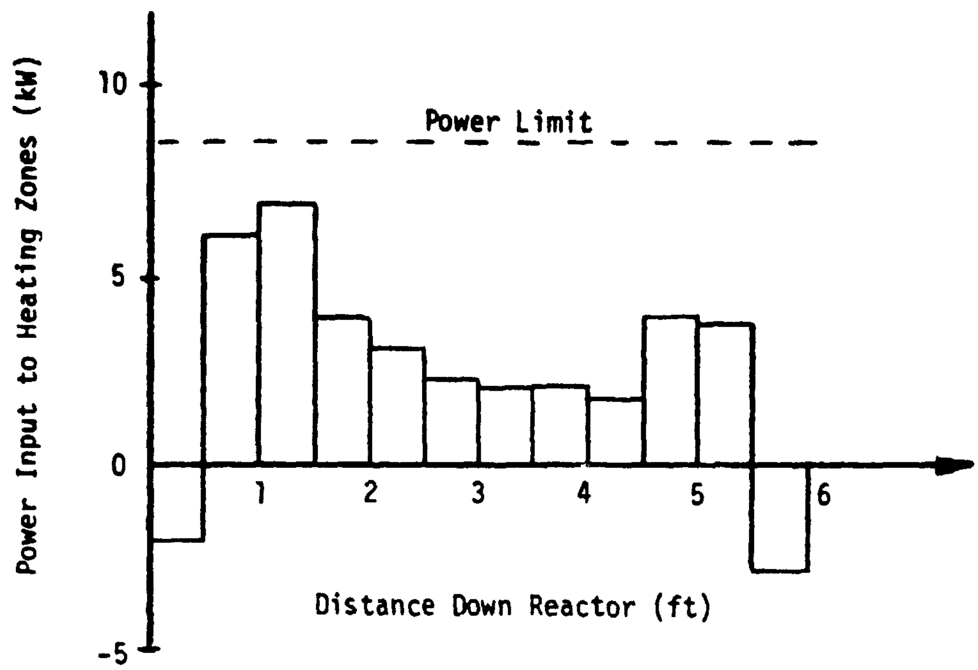


Figure 6 Power Inputs to the Heating Zones - EPSDU Reactor Simulation

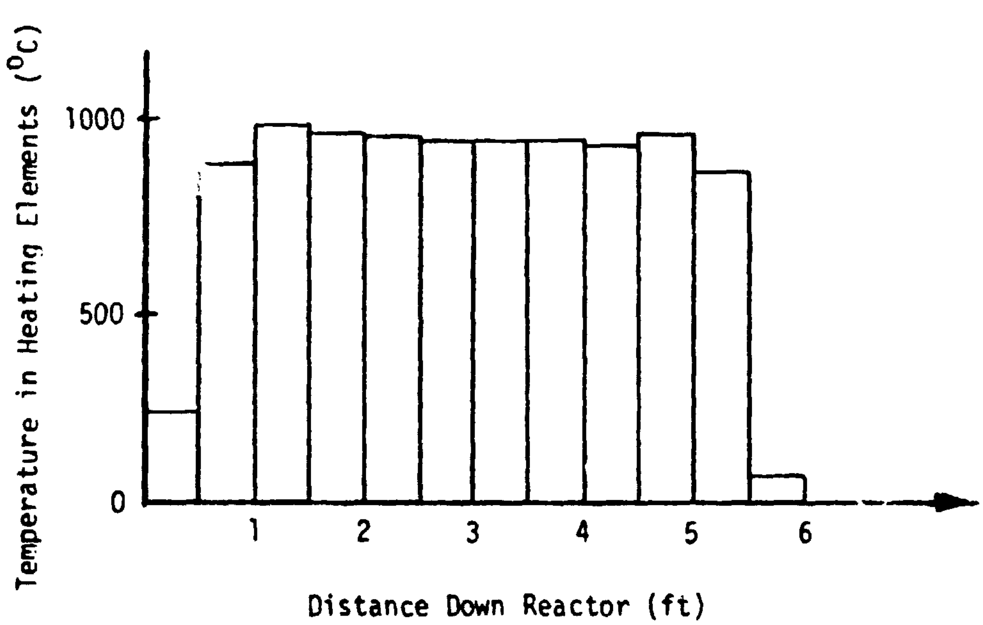


Figure 7 Temperatures of the Heating Elements - EPSDU Reactor Simulation

Crystal Growth Experiment Using Free-space Pyrolysis Powder

A crystal growth experiment was conducted by Kayex Corporation on October 20, using silicon powder produced by the free-space reactor. The primary objective was to establish the feasibility of growing an ingot by introducing silicon powder directly into the crystal grower.

A powder-feeding device based on an auger was designed and fabricated to transfer powder directly from a storage drum to the crystal grower. A CG 2000 RC puller was used. The drum-auger assembly, containing silicon powder from free-space reactor Run 13, was connected to an inclined quartz tube entering the crystal grower through a top port. A ball valve was used to isolate the crystal grower. A ceramic-disk plunger was provided to clear the quartz tube in case of blockage.

Powder was fed to the crucible by manually rotating the auger after applying power to the puller. Powder feeding was satisfactory, however, considerable blowing of the powder in the furnace chamber was observed. This uncontrolled blowing of fine powder caused arcing problems whenever the powder got in between the crucible and the graphite heating elements. Powder melting was effective. Since the power input had to be limited to 50 to 60 kW due to arcing, and the crucible could not be lowered into a higher temperature zone, the feeding and melting steps were rather slow, taking almost 6 hours to establish a stable melt of approximately 6.5 kg of silicon. The melt was relatively free from silicon monoxide; however, a glassy slag formation observed in the melt was not explained until completion of the experiment. When the equipment was disassembled at the end of the run, it was realized that the ceramic plunger disk (1.25" dia. x 0.25" thick, containing approximately 7% boron), designed by Kayex, had accidentally fallen into the melt.

Crystal pulling was hampered by several factors. Crucible rotation had to be stopped due to arcing problems. Fluxing and devitrification of quartz crucible, caused by the dissolved ceramic disk, resulted in quartz particles migrating towards the seed and affecting crystal growth. Kayex was able to pull polycrystalline ingots of 1 inch and 4 inches in diameter and 3 inches long. Due to the heavy accidental doping, resistivity of the polysilicon ingots was low, typically 0.04Ωcm, P type.

On November 20 a second powder-melting and crystal-pulling run was conducted by Kayex Corporation using silicon powder produced by our free-space reactor. The powder from our Run No. 17 was transferred into the stainless-steel transfer-drum directly without exposure to the atmosphere. The drum was initially kept under a nitrogen purge and finally under an argon purge. This drum, containing an auger, was connected to Kayex's CG2000 RC crystal puller. The ceramic plunger, which caused contamination problems in the previous run, was eliminated. The crystal-puller furnace was equipped with a new graphite hot zone. A quartz ring was used to shield the annular space between the quartz crucible and graphite heater. This shield prevented powder from getting into the annular region and thus eliminated arcing problems experienced during the previous run.

Powder feeding and melting operations were effective. The feed and melt rate was approximately 3.2 kg/hour, and there were indications that this rate could be considerably increased. Attempts to pull a single crystal ingot were hampered by the presence of a contaminant "scum" floating on the melt surface. This contamination seemed to originate from powder that had reacted with some component of the furnace atmosphere. When the gas flow around the crucible was disturbed during attempts to remove the contaminants, additional powder with contaminant fell into the melt. The bulk powder did not appear to contribute to this contamination.

Kayex was able to pull a polycrystalline ingot 3 inches in diameter x 4 inches long. The resistivity of this poly was measured to be 55Ωcm, P-type. This resistivity value is excellent and indicates that our free-space pyrolysis powder is of high purity.

1.6.2 Melting/Consolidation

The design and development effort necessary to obtain a reliable melter for EPSDU involves UCC and sub-contractor (Kayex) effort.

1.6.2.1 Melter System Subcontract

Kayex Corporation is developing the silicon melter system for EPSDU. The silicon consolidation scheme is based on melting the powder in a quartz crucible and dropping molten silicon shot from the crucible bottom into a cooling tower where the shot is solidified. The goal is to design and build a melting/consolidating system suitable for installation in the EPSDU.

During the first quarter of this contract, Kayex conducted a theoretical study of the shotting process, met with Union Carbide concerning the theoretical approach, began design of the prototype assembly concentrating on long delivery time items, purchased several items, and conducted ten small scale melting runs to demonstrate the feasibility of the shotting concept and to obtain process operating experience.

During the second quarter, Kayex determined an appropriate crucible orifice size based on small-scale melting run results, devised a system of process controls, and neared completion of system design and part ordering for the shotter.

The current quarter project goals have been to complete system design and part ordering, to assemble the system as parts arrive, and to finish the small-scale melting runs.

Process Controls

Sensotec Corporation is producing most of the process control equipment to be used including the load cell, the pressure transducers, and all amplifiers and digital readouts. The Sensotec Sensors were deemed to be the best available, so it was decided to use Sensotec equipment for the entire system to make the system as homogeneous as possible.

Infrared light sources and photocells were ordered for detecting both shot flow and powder level.

A shaft rotation magnetic rate sensor will be used to monitor auger feedrate and to send an output signal to a DVM in the control panel.

Flow switches and alarms are used to monitor cooling water flow rates and a chart recorder to monitor important process variables such as:

- The shotting flowrate by monitoring the photocell output in the lower control mounting chamber.
- The temperature of the hot zone by monitoring the optical pyrometer output in the furnace tank.
- The net melt weight (i.e., the total melt weight sensed less the gas pressure difference) by monitoring the output from the differential amplifier which subtracts gas pressure difference from gross melt weight.

The powder level sensing method has been demonstrated in a laboratory simulation with an IR light emitting diode and a standard silicon phototransistor.

Design Review

On September 10, Kayex and Union Carbide representatives met at Kayex's facility in Rochester.

The following issues were discussed/resolved:

- Transfer of powder from a drum to the feed hopper could be achieved by using a hand-operated mixer attached to the drum. Concern was expressed that the driving gas may channel through the powder since the powder has low density.
- A handwiper would be adequate for observing powder level in the hopper.

- Union Carbide inquired about the advantage of induction over resistance heating since we do not couple directly to the melt. Kayex responded that induction heating is quicker to cause a temperature response for a given change in power applied. This is because in resistance heating, the power applied must heat the heater elements which must, in turn, heat the crucible support by radiation and convection which, in turn, heats the melt. The heater elements also heat the furnace walls through the insulating heat pack. In induction heating, the power applied (after a slight loss in the coil) directly heats the graphite susceptor which, in turn, heats the melt.
- Union Carbide approved the use of a fan with a filter for introducing air to the control cabinet and a louver for an outlet to make the cabinet "dustproof".

Equipment Design and Purchasing

The following paragraphs discuss the components for the melter and shotter.

Gaskets

A silicon rubber soft cylindrical gasket will be used for sealing between the furnace tank and the drop tube. The gasket is soft enough to compress under an increasing load without producing a significant force opposed to the downward force of the hot zone (which would affect the weight readout).

Graphite Elements

Two pre-purified graphite susceptors were ordered from Weaver as well as a graphite insulating piece.

Two graphite rupture discs, rated to burst at 15 psig, gaskets, supp rt sleeve, and a deflecting elbow were also placed on order.

Auger Design

A preferred seal design for the auger system was approved by Union Carbide, and ordered.

The extension for the auger hopper, expands the hopper capacity to 60 gallons while maintaining 10 psi capability, was contracted to the auger manufacturer.

Quartz Drop Tubes

Five quartz drop tubes for channeling powder from the auger outlet to the crucible have been ordered. Five quartz radiation hood supports for supporting the molybdenum radiation hood and for use as a crucible cover have also been ordered.

Recorders

A Leeds and Northrup Speedomax Mark III chart recorder with three channels and a 15-inch per hour chartspeed has been obtained.

Pneumatic Powder Transfer

The pneumatic powder transfer method and a tentative design for the powder drum, hopper, and connecting hose were established after discussions between Kayex and Union Carbide. Kayex will use a small pump on the hopper gas outlet to help pull the powder into the hopper and reduce the pressure requirements on the powder drum. Since the pressure transducers will be arriving too late for use in starting up the system, two mechanical pressure gauges have been obtained for this purpose.

Silicon Chunks

A sample bag of 5 kg of virgin, off-specification polycrystalline silicon "nuggets" was obtained from Smiel, but were too fine to etch and use for melting. "Chunks", ordered from Silicon Casting, will be evaluated for use in melting.

Induction Heating System

Design drawings for the induction heating system were sent by American Induction to Kayex for review and approval.

Weight Ring

The design for the weight-ring legs was changed so that the leg resting on the load cell will be anchored in the weight ring and will be of fixed length. The other two legs can be adjusted from outside the furnace tank.

A housing for the load cell has been ordered. This will mount on the upper flange of the lower control mounting chamber. Screws for adjusting the weight ring have been ordered, these will mount on the upper flange of the lower control mounting chamber.

Crucibles

Three crucibles without nozzles have been ordered from Heraeus Amersil. These will be modified when an appropriate nozzle design has been chosen, based on preliminary run results using the five round-bottomed crucibles with nozzles tapered from 2 to 1 mm. Rotameters for all water flow circuits have been obtained as have water flow switches.

Helium

Compressed helium cylinders (213 cubic feet at 2200 psi) will be used for the shotter system purge gas since liquid helium is not available. It is roughly estimated that one shotting run using one crucible full of silicon chunks would require at least two cylinders of helium. One cylinder would be required for the furnace tank whose volume is 7 cubic feet and one for the drop tube whose volume is 2.5 cubic feet. The drop tube will require a much greater flow rate than the furnace tank, since a steady gas stream is needed to cool the falling shot. One for each chamber will provide 30 furnace tank volumes and 85 drop tube volumes of gas throughput. A blanket order for 50 cylinders of Linde industrial grade (99.996% pure) helium has been placed.

Equipment Deliveries

The shotter framework and platform were delivered during October on schedule. Other parts received include the furnace tank, baseplate and lid, graphite susceptors and insulating blocks, molybdenum radiation hoods, and gas valves and regulators. In addition, we received the alumina radiation shield and hot-zone support tubes, the steel elbow and flange for deflecting the rupture disc gas flow, the auger support assembly, two 11 gpm flowmeters for capacitor bank and generator water flows, five round-bottomed crucibles with nozzles from QSI, a 5 kg sample of polycrystalline, 0 to 5 ohm-cm, p-type silicon chunks from Silicon Casting, and six cylinders of 213 ft^3 each of helium at 2200 psi.

The induction heater system was designed and supplied by American Induction.

Assembly and Scheduling

Riggers were contracted to assemble the shotter platform, supporting structure, and ladders and railings. The shotter structure components have been bolted together so that it can be disassembled and shipped when necessary. The railing around the primary platform has been modified to include a sliding gate to allow easy passage of material onto the platform when using the crane. A secondary platform has been mounted 7 feet below the primary platform to allow for easy access to viewport window, and to facilitate working on sections below the primary platform.

The furnace tank baseplate has been drilled and tapped for the alignment brackets for the alumina radiation shield tube, and these brackets have been machined and installed. The furnace tank, baseplate, and lid have been mounted and leveled on the shotter's primary platform.

The construction phase should extend 2 months beyond the originally projected deadline because of the failure of several key vendors to meet delivery dates.

The beginning of the debug and test phase will be delayed by 2 months because of the late completion of the construction phase. The process development phase, scheduled to begin in November, will be delayed by 2 months for the same reason. It may be possible to make up this time in the 10 remaining months of the project, especially during the process development phase.

Small Scale Runs

The last small scale melting run was conducted in October with silicon powder and chunks. The chunks were on top to prevent the powder from rising out of the crucible. The purpose of this run was to determine if previous powder melts did not produce shot (unlike chunk melts) because of insufficient head from low-density powder. During the run, the old cracked graphite created extreme arcing problems, cracked further, and damaged the crucible wall as well as the outer, quartz, argon-containing purge-tube. The heater had to be powered down prematurely, but nearly half of the silicon melted without producing shot. It is inconclusive whether there was insufficient melt head to force shot passage.

1.6.3 Fluid-Bed Development

This development program includes all analytical, experimental, and design effort associated with developing a fluid-bed reactor as an alternative or backup system to the free-space reactor.

1.6.3.4 Fluid-Bed Pyrolysis (PDU)

This work item consists of all effort associated with the design, fabrication, and testing of an experimental unit to establish design data for an EPSDU-scale system.

PDU Fabrication/Assembly

The design of the PDU was completed in October. All fabrication work was completed in December. The expanded head, fluid bed reactor pipe, and particle separation boot are connected and mounted on the support structure. All valves, process lines, and most of the instrumentation are installed and piped.

The particle withdrawal section from the boot to the product hopper was assembled and tested. Silicon particles were successfully transferred into the product hopper by gravity or diverted pneumatically with a high-velocity hydrogen stream to a small sample hopper.

About 300 lb of high-purity silicon was ground and screened. About 45 lb will be acid cleaned and etched. This will be the initial charge for the fluid bed on start-up.

Feeding of silicon seed particles into the fluid bed will be controlled by a small screw auger feeder which was assembled and tested. For every 180 degree rotation of the auger, 2.0 $\pm$ .1 grams of silicon was passed from the feeder. The amount of seed required to sustain continuous operation is calculated to be about 14 grams per hour.

Operating procedures for the fluid bed unit were written. This includes detailed step-by-step instructions for initial start-up and operation of the fluid bed.

Remaining work includes installation of thermocouples, resistive heaters, capacitive electrode heater assembly, insulation reactor outer walls, product hopper to boot, sample hopper from the product withdrawal line, and a few remaining instrument and air lines. This work should be completed in January 1981 and check-out will then be started.

Operation of the two power generators and matching transformer networks required for electrical heating was checked out using a dummy load.

The electrode assembly has been assembled. The stepping motor worked satisfactorily and the control position indexing switch has been calibrated to indicate electrode spacing. The ground electrode shaft is not acceptable because of surface defects. There is a possibility that the O-ring will not seal. Also the location of the electrode element crossarm with respect to the power electrodes provides a shorter electrical path than the electrode elements

in some positions. A new ground electrode is being fabricated from centerless ground and polished shafting with the crossarm location changed to be in line with the quartz tube insulators of the power electrodes. This eliminates the potential electrical path from crossarm to power electrode if the silicon bed height goes above the crossarm.

The operating procedure has been written for the direct electrical heating of the silicon bed. This includes a description of the electrical equipment and electrode assembly and their operating characteristics. A step-by-step startup and load balancing method is outlined and use of one or both electrode circuits is discussed.

Capacitive Heating

A comparison of the electrical characteristics and equipment requirements for capacitive heating vs arc-discharge heating was completed and is summarized in Figures 8 through 13 and Table IV. This indicated a need to know the limits for the capacitive heating mode. The maximum field strength which may be applied is determined by the breakdown voltage for the gas in the silicon bed, and the magnitude of this voltage was not known. The maximum input power without breakdown occurring also was not known.

A series of laboratory experiments was conducted using a flat parallel-plate test cell (Figure 10) with a uniform electric field to determine the power density in a settled silicon bed as a function of field strength and frequency. The bed volume impedance as a function of frequency was also measured. The leading phase angle varied from about 40^{o} at 10 kilohertz to 87^{o} at 1 megahertz and the bed has a very high dielectric constant. Tests were conducted with the aluminum electrode plates contacting the bed and with a 3 mil thick mylar film between the plates and the bed to simulate a dielectric coating. The electrical properties were changed only marginally by the mylar film. In one set of tests there was a slight increase in power density and in another test series a slight decrease. Above 100 kilohertz the power denisty increased only slightly with frequency. The power density at a field strength of 600 RMS volts/cm and 100 kilohertz was o.15 watts/cm^3.

Tests were conducted with beds of metallurgical-grade silicon of different particle sizes and semiconductor-grade silicon of wide particle size distribution having a mean particle size of 222 microns. It was found that the volume impedance of the bed is independent of particle size and grade of silicon.

A parallel-rod test probe (Figure 13) was fabricated to measure the breakdown voltages of settled silicon beds in air at room temperature. The breakdown voltage was very dependent on particle size and decreased rapidly with increasing particle size. The breakdown voltage also increased with frequency up to about 60 kilohertz and then stayed about constant at higher frequencies. For the semiconductor-grade silicon bed of wide size distribution ($\bar{d}_p$ = 222 microns) the breakdown voltage was 1890 RMS volts/cm at 100 kilohertz. Comparing this to published values for air in a uniform field, the silicon bed reduces the breakdown voltage by slightly more than one order of magnitude. For the case of hydrogen in the fluid bed pyrolysis reactor at normal operating conditions, the maximum continuous field strength is estimated at 500 RMS volts/cm with the power density being 0.11 watts/cm^3.

1.6.4 Quality Control

This task includes all activities associated with the development of quality control equipment, techniques, and procedures for use during EPSDU operations.

1.6.4.1 Silane Analysis - Slim Rod Deposition

Polycrystalline silicon rods have been routinely grown in the laboratory slim-rod deposition reactor. Using undoped, pure silane feed gas depositing onto a 3 mm square x 150 mm long seed rod, a polycrystal rod of 8.25 mm square was grown in 14 hours. The average deposition rate in successive runs was nominally 1.0 gram/hour at 850°C deposition temperature. The bulk resistivity of the deposit was 253 Ωcm with a variation of 16 Ωcm along the rod as measured with a colinear four-point probe. Surface quality of the deposit was good. The growth was uniform over the length of the rod with a rounding off at the ends. No growth of silicon was evident on the water-cooled carbon chucks and, as shown in Figure 14, the end of the original substrate could be removed intact from the chuck. This provides a convenient, concentric mounting point for subsequent zone refining.

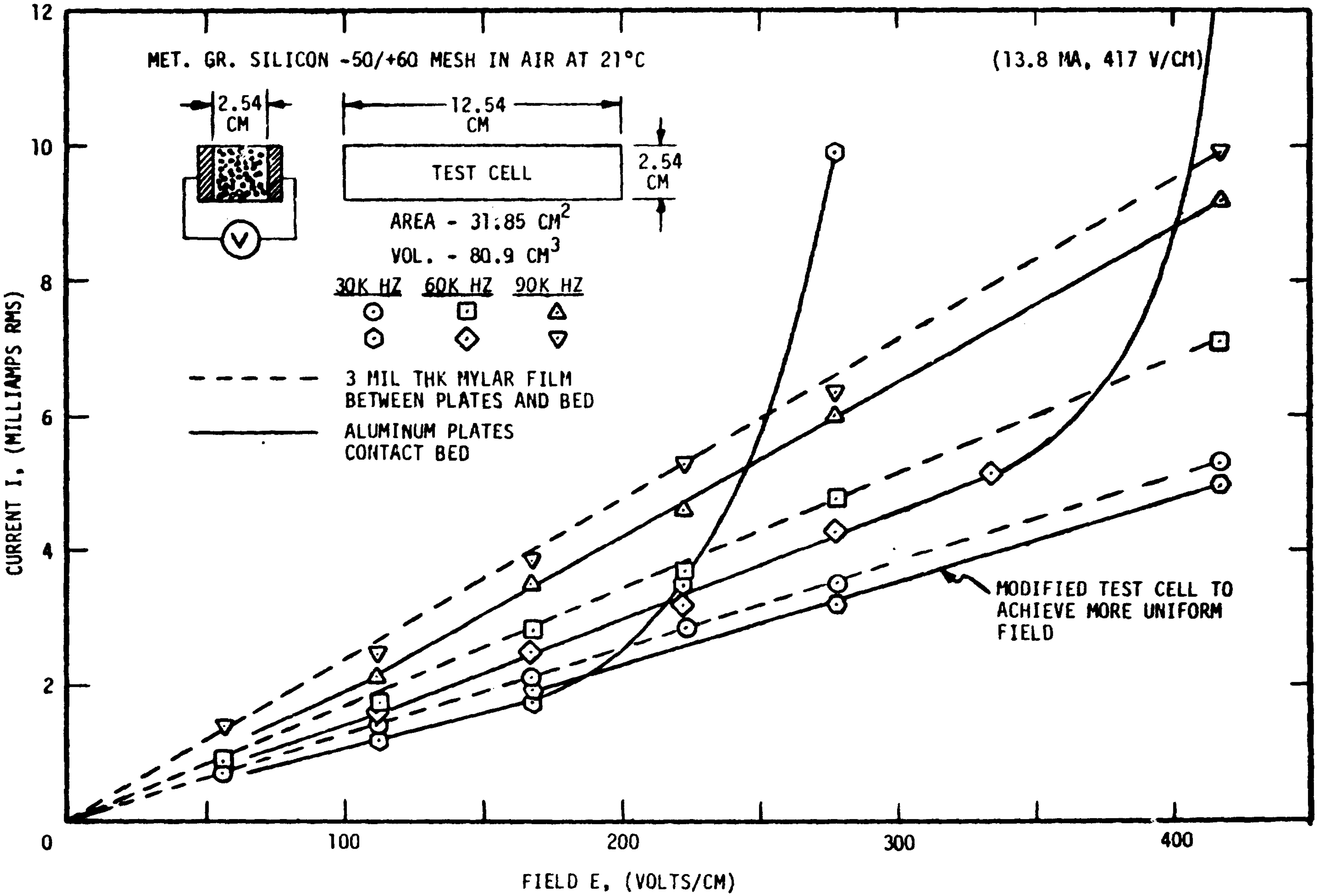


Figure 8 Electrical Properties of Static Silicon Particle Bed; Current vs Field Strength

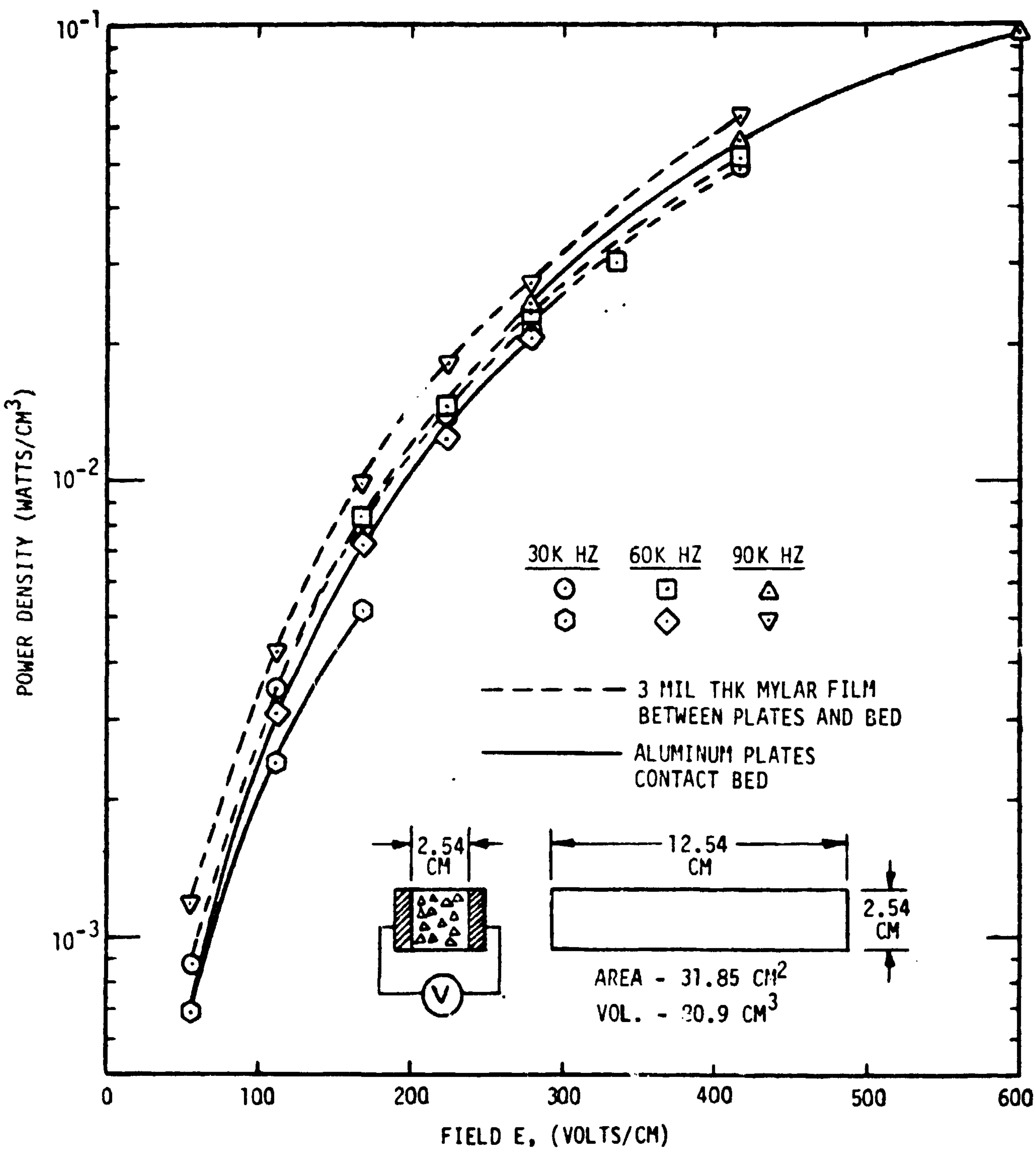


Figure 9 Electrical Properties of Static Silicon Particle Bed; Power Density vs Field Strength

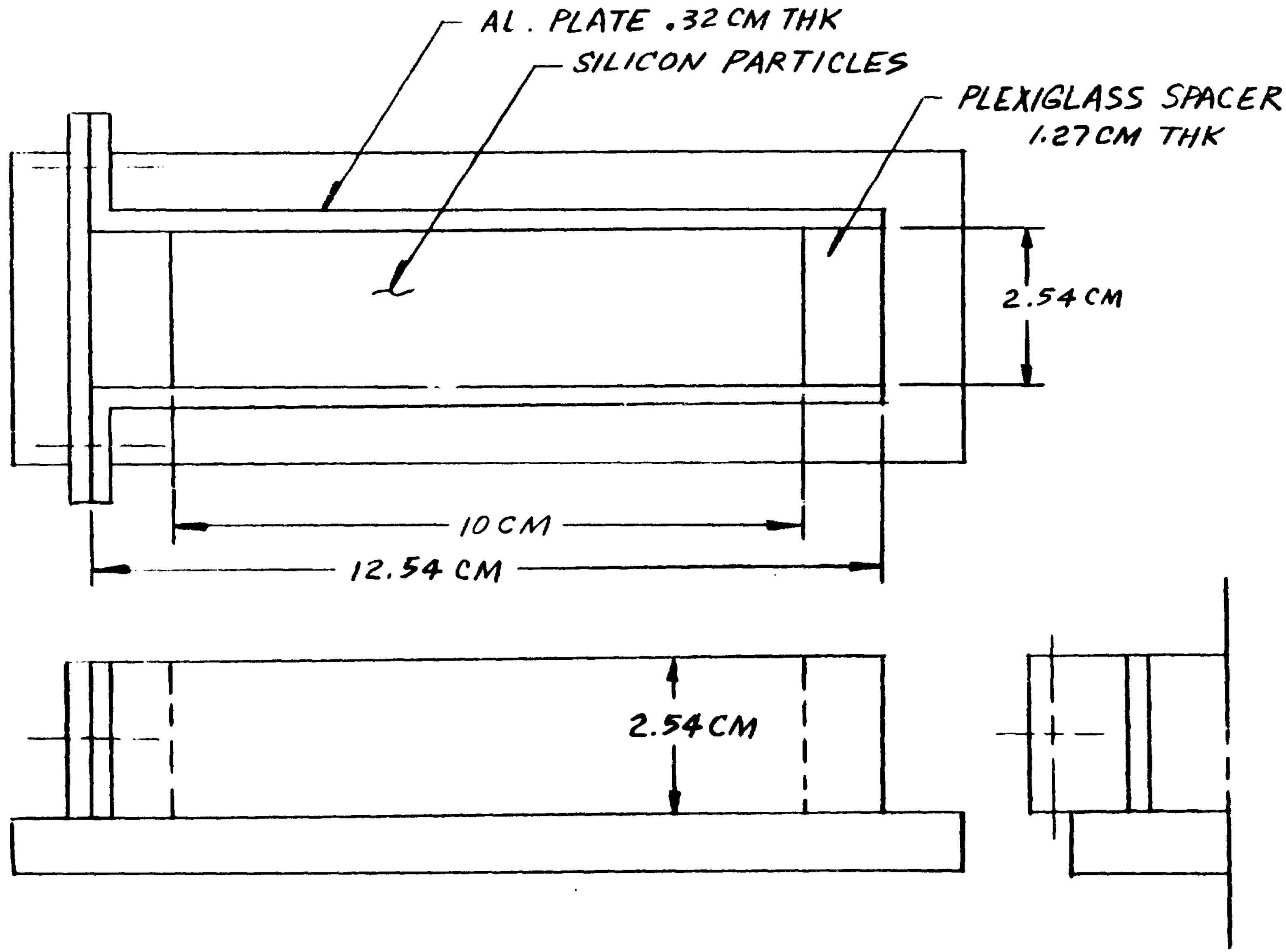


Figure 10 Parallel Plate Test Cell

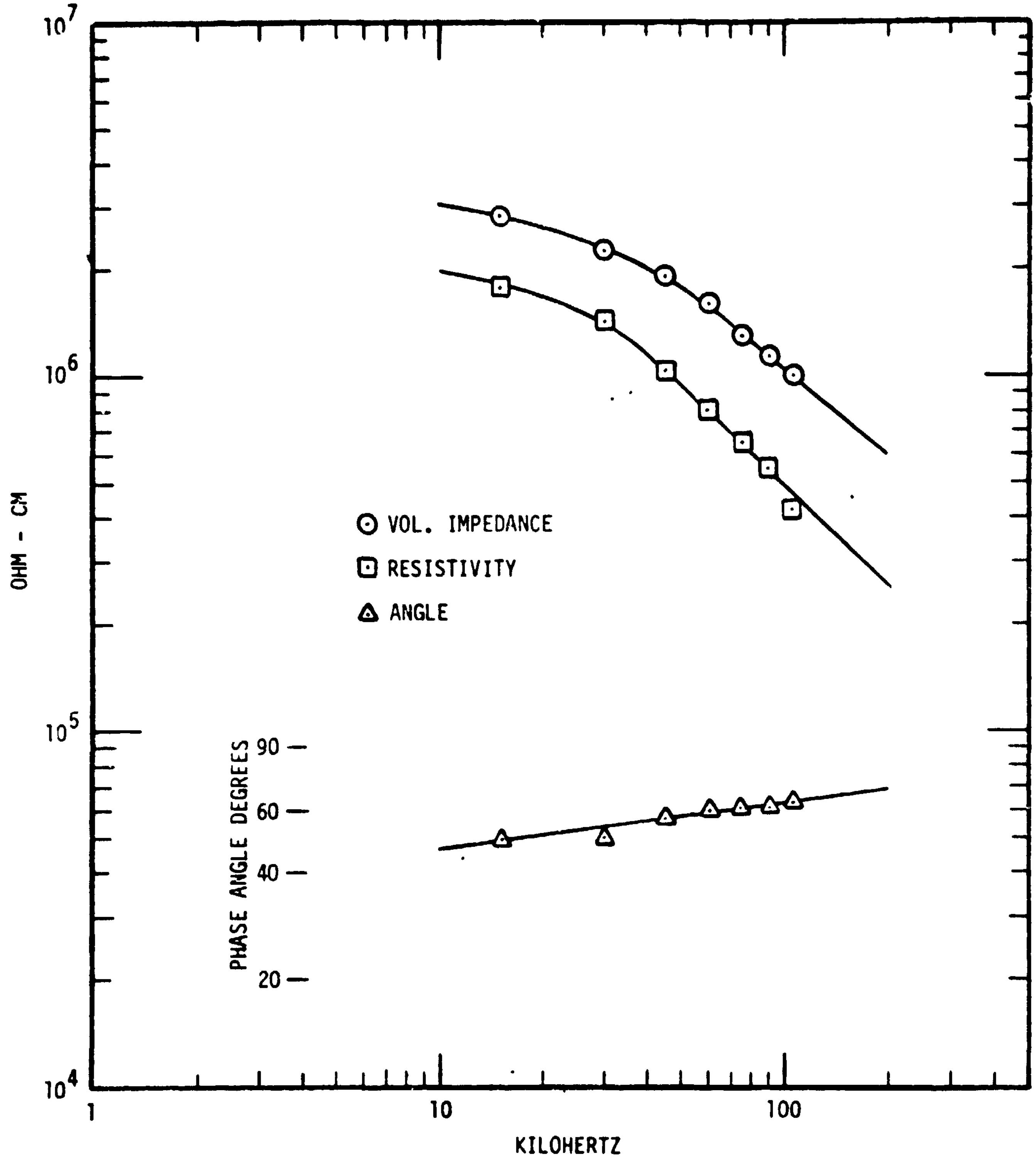


Figure 11 Electrical Properties of Static Silicon Bed; Resistivity, Phase Angle vs Frequency (M-G Silicon)

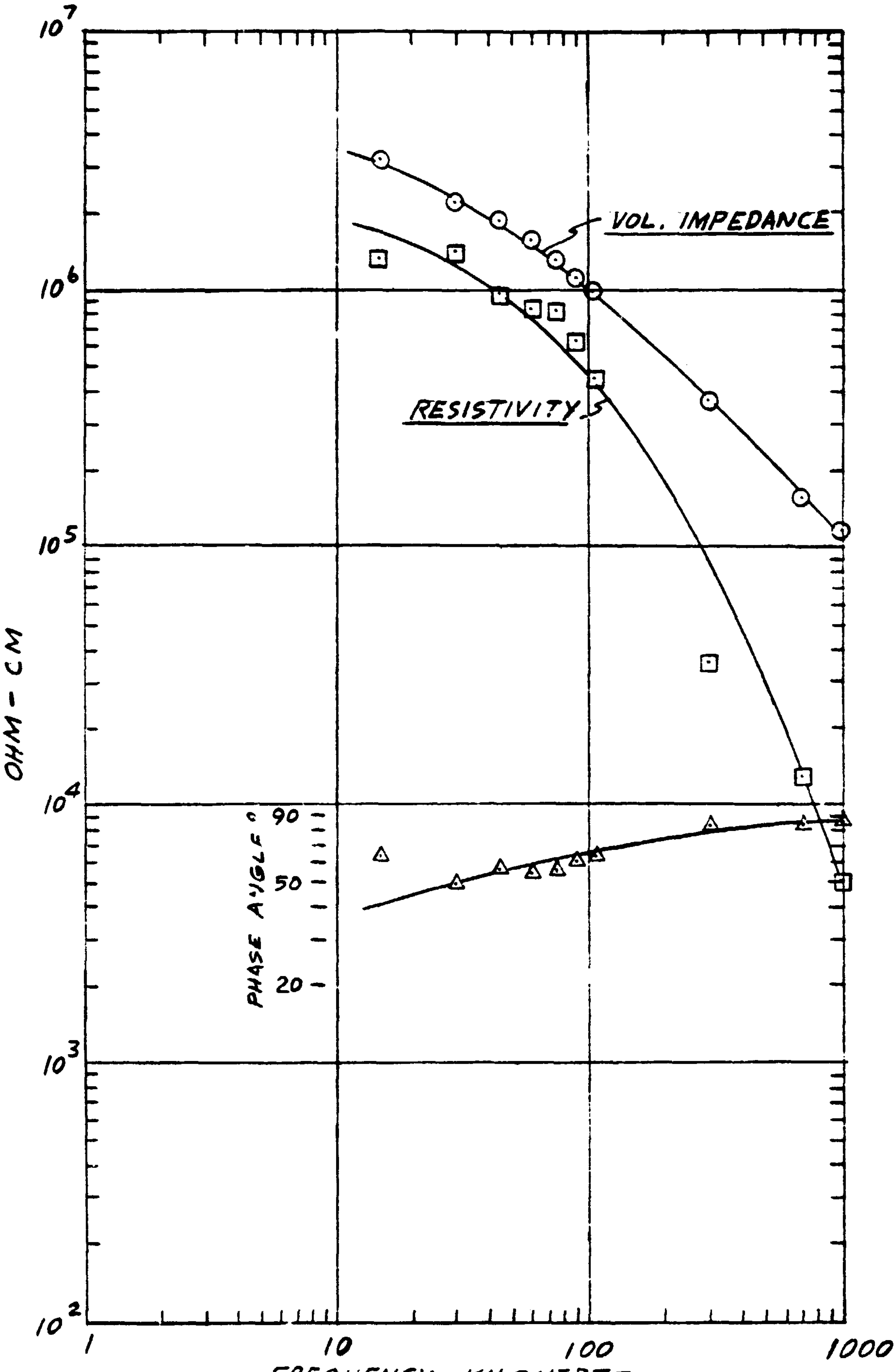


Figure 12 Electrical Properties of Static Silicon Bed; Resistivity, Phase Angle vs Frequency (Semiconductor-Grade Silicon)

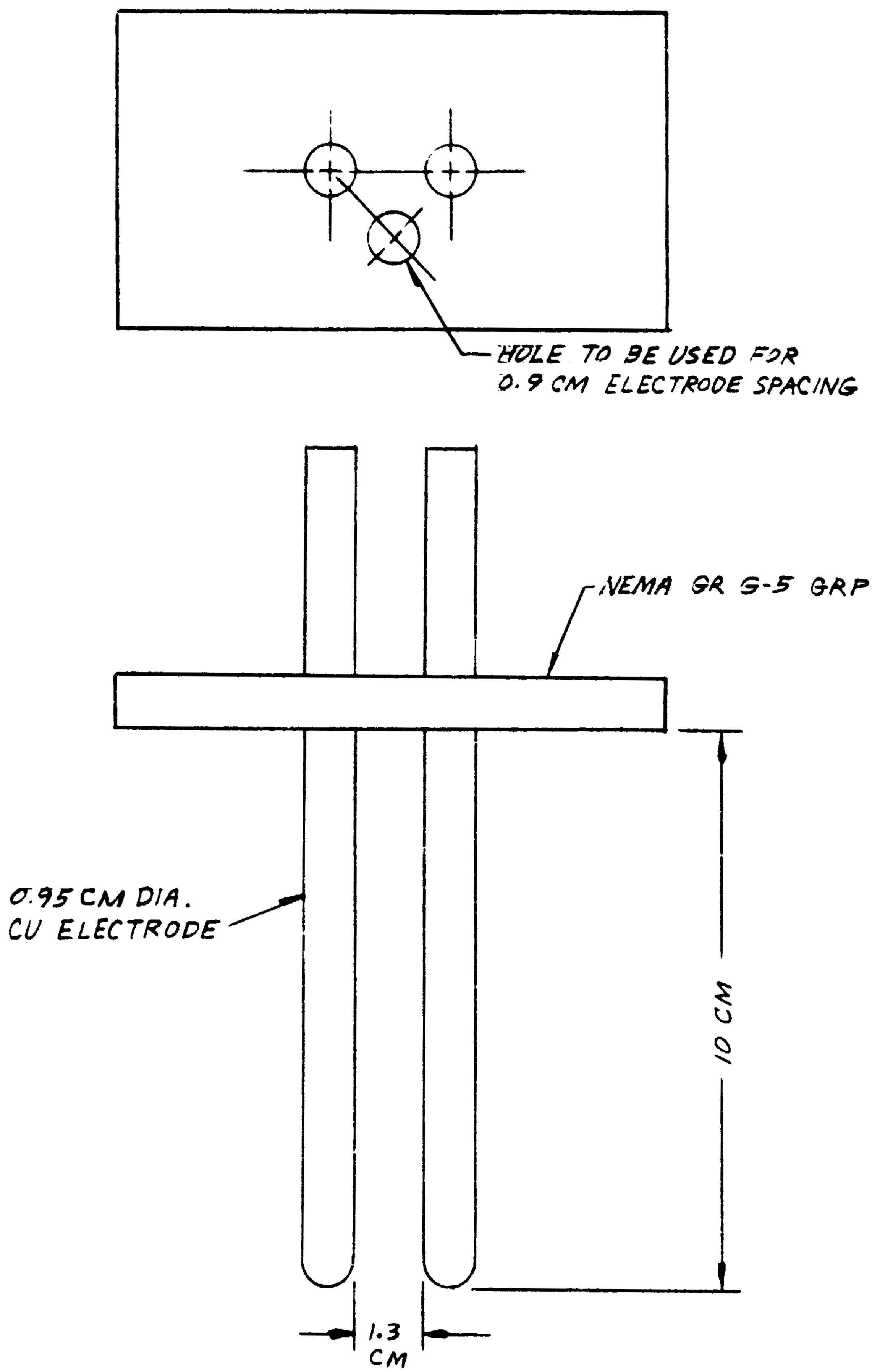


Figure 13 Probe for Measuring Breakdown Voltage

TABLE IV CAPACITIVE HEATING TESTS; OBSERVED BREAKDOWN VOLTAGE

TEST NO	SILICON	ELECTRODE TYPE	GAS	PRESSURE (ATM)	TEMP ^{o}C	$\frac{U}{U_{mf}}$	BREAKDOWN VOLTAGE RMS VOLTS/CM	
							OBSERVED IN BED	CALCULATED FOR GAS IN UNIFORM FIELD
26-2	MET. GR. -20/+230 MESH $\bar{d}p$=210μ	PAIR - 1" DIA (d = 2.54 cm)	He	0.07	20	2	129	278
26-29		PAIR - 1" DIA (d = 1.59 cm)	N_2	0.11	20	0	310-600	3379
26-84		PAIR - 1" DIA (d = 1.59 cm)	He	0.49	20	1.3	335	1200
13	SEMI-CONDUCTOR -80/+120 MESH $\bar{d}p$=187μ	.5" DIA ELECTRODE TO DISTRIBUTOR (d = 3.81 cm)	N_2	1	20	2.6	26	18556
16		.5" DIA ELECTRODE TO DISTRIBUTOR (d = 5.4 cm)	N_2	1	400	6	145	8477

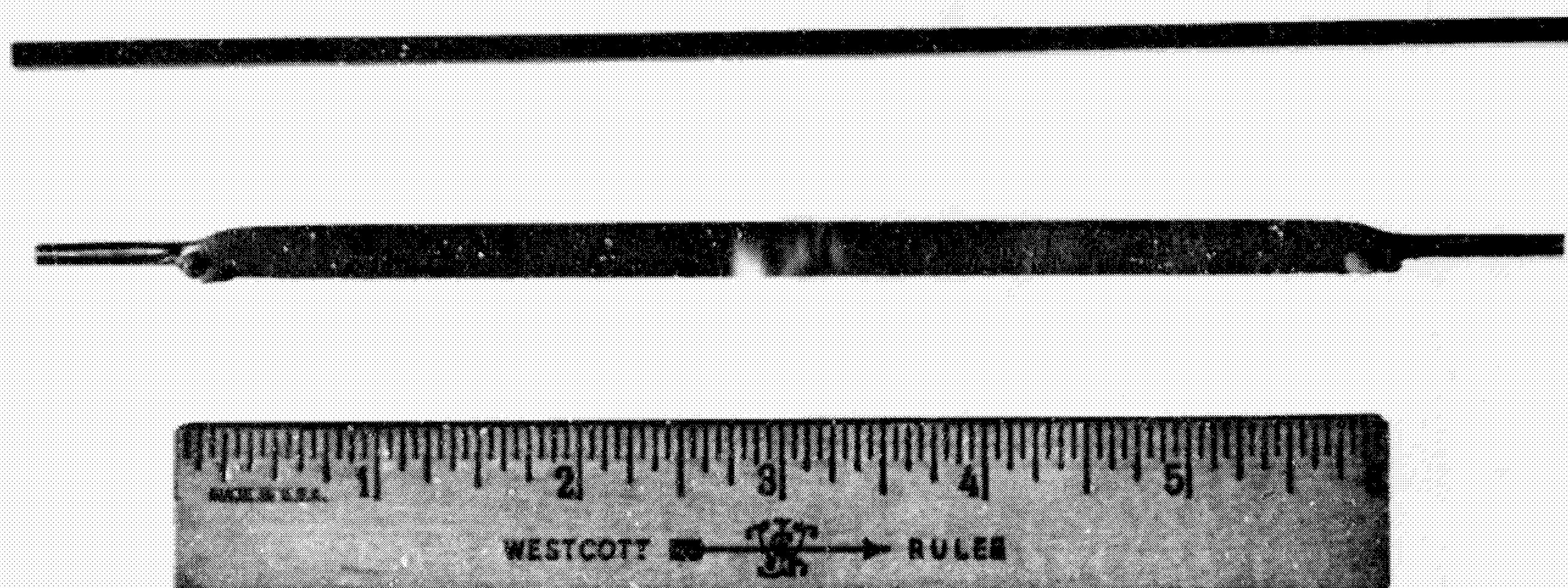


FIGURE 14 SLIM - ROD SILICON DEPOSITION

To complete the sequence as a quality control analytical method, the polycrystal rods will be subjected to multi-pass zone refining and the resistivity profile measured along the resulting crystal. The fresh silicon deposit is intimately mixed with the seed during zone melting. The planned ratio of fresh silicon to seed is >5:1. The quality of the first three polyrods grown this period define the baseline silane quality.

During this period growth runs were not performed as the major activity was to identify and resolve sources of contamination in the reagent gas supply system, portions of which are common to both slim-rod and epitaxy reactors. We previously established all parameters for routine uniform growth of silicon on heated seed rods from silane and further runs at this point, while adding to our experience factor, would have diluted our efforts toward resolving the silane purity problem.

The final experimental work using the slim-rod pyrolysis reactor was completed in December. A series of runs was made using silane having controlled concentrations of diborane or phosphine after a series of three runs using undoped silane were made to establish baseline quality. The objective of this program was to develop a technique which could be used to convert a sample of silane into a desireable form of silicon of equivalent quality which could be analyzed by standard techniques used by the industry; namely multipass float zone refining and resistivity profiling.

Three 14-hour growth cycles were made using high-purity silane gas. This gas had previously been used in the epitaxial reactor where film resistivities of >150 Ωcm N-type had been measured indicating that the source gas was of high quality. The polysilicon growth onto the 3 mm square float-zoned single-crystal seed rods was smooth and uniform. The average rod diameter was 8.25 mm; the new silicon constituted 87% of the esulting rod. The resistivity of the rod, measured using a 4-point probe, was 250 Ωcm. This indicated that no gross impurities were present although more accuracy would require conversion of the polycrystal silicon rod to single crystal.

Three growth cycles were made with phosphine and three with diborane dopant gas mixed into the silane at various levels from 2 to 20 ppb. The dopant was injected into the silane after diluting the 50 ppm source gas with H_2. Again,

14-hour growth cycles yielded 8.8 mm diameter rods containing 87% new silicon. The electrical resistivity of the grown rods will be measured and compared with the dopant concentration in the feed gas to provide a "capture efficiency" in the silane-to-silicon deposition.

1.6.4.2 Silane Analysis - Direct Measurement

Diborane/Silane Vapor-Liquid Equilibria: The vapor-liquid equilibria (VLE) of a diborane/silane mixture was measured at 40°C and 300 psi, the planned operating point of the EPSDU silane distillation column. The relative volatility of silane over diborane was measured as 2.03 ±0.07 at a diborane concentration of one percent. This helps to substantiate the operating basis of the EPSDU silane column which is designed to separate the more volatile silane in a high tray-count column. Tests for thermodynamic consistancy and confirmation of the VLE model used for the SiH_4/B_2H_6 system is planned.

The experimental technique utilized in this determination involved condensing a mixture of silane containing one percent diborane into a liquid-nitrogen-chilled equilibrium cell. The cell was cooled to -40°C where it was maintained with mechanical agitation to assure equilibrium conditions. Through a plumbing system submerged in the -40°C bath, samples of vapor and liquid were withdrawn. The samples were analyzed by passing the vapor (or vaporized liquid) through a small quantity of high purity water containing a pinch of curcumin. The amount of boron trapped was measured photometrically using a UV spectrophotometer. Several determinations were made on different days with good agreement among them. The data have been sent to the UCC physical properties measurement group at South Charleston for verification. An evaluation for thermodynamic consistency shows the system to be nearly ideal.

The original EPSDU column design was made assuming ideal gas rules, and hence the design basis has been confirmed; no further modeling or changes in column design are required.

1.6.4.3 Silane Epitaxy

The purpose of this work was to develop a rapid analytical technique for qualitatively determining the electronic quality of silane produced by EPSDU prior to committing the silane to the decomposition reactor. The technique is based on forming an epitaxial film of silicon from the silane sample onto

an N - or P-type single crystal substrate wafer and measuring the film resistivity. Net donor or acceptor levels can be inferred and a minimum resistivity specification can be established for silane suitable for conversion into product silicon.

The air-cooled epitaxy reactor was operated to produce nominally 15 μm films on 30 mm diameter, 60 Ωcm P-type wafers from undoped silane. Wafer cleaning techniques were defined to substantially eliminate crystal imperfections. Several sources of contamination were identified and corrected including a "dirty" HCl regulator, and a H_2 purifier element. The quality of epi-films grown routinely this period was quite good. Epi-film thickness was measured non-destructively using differential interference contrast microscopy to measure stacking fault dimensions which are directly related to film thickness as taught by ASTM 143-73. A film thickness calculated from wafer weight gain is being compared with the optically determined value as a potentially quick method of thickness determination. To provide sufficient stacking faults, each fresh wafer was probed with the four-point probe to inflict modest surface damage prior to epitaxy growth.

The four-point probe utilized for measurement of film resistivity was checked using a fresh wafer with an independent four-point probe and found to read within one percent. All resistivity measurements on epitaxy films are made with the specimen located in a light-tight box to avoid photovoltaic effects. An adjustment is provided for compensating for epi-film thickness. Conductivity type is determined by the rectifying technique for bulk specimens and by the thermoelectric method for the epi-film.

In determining the "base line" resistivity of the undoped silane supply, multiple runs were made in a single day using, as near as possible, identical wafer handling and reactor operational techniques. After cleaning the fresh wafer in an ultrasonic detergent bath, rinsing with electronic-grade methanol and Type I water and clean-air drying, the specimen is loaded onto a silicon-coated graphite susceptor and placed into the continuously purged epi-reactor tube. An H_2 purge while heating to 1100°C is followed by a 10-minute HCl etch and cooling to 1050°C. At that temperature a 20-minute deposition period with 0.5% SiH_4 in H_2 produces a 15 μm film. Electrical measurements are made on the cooled wafer.

A low average film resistivity with large run-to-run variation was observed when nominally 15 μm films were grown from a single source of silane on 60 Ωcm P-type wafers. Various trouble-shooting activities showed that improvement in resistivity values were obtained when:

- The ultrasonic wafer pre-cleaning bath was cleaned at prescribed intervals.
- Several fittings contaminated with C-100 thread lubricant, were identified and cleaned.
- The silane, hydrogen, and HCl supply lines were acid cleaned, rinsed, and dried.

A significant step increase from approximately 40 Ωcm to over 100 Ωcm was obtained when an alternate silane supply cylinder was used. Both cylinders were presummably "electronic grade" with a resistivity of >100 Ωcm when manufactured. The second cylinder had about 6 months shorter shelf time prior to use. The reasons for low purity in the first cylinder have not been established. The second cylinder will be used in all subsequent work. Even after these sources of contamination, which resulted in higher average resistivity values, were identified and resolved, there was still a significant variation in the measured epi film resistivity.

A series of runs was made varying the duration and concentration of the HCl etch. HCl is potentially the dirtiest of the gases used in the epitaxy test, thus, the concentration and duration of the pre-etch could have significant impact on final film quality. As listed in Table V, the surface quality of the film and the resistivity variation across the wafer improved when the HCl etch was reduced in concentration (flow rate) while etching for a slightly longer period. The H_2 carrier flow was constant at 6 liter/min, through the 38 mm ID reactor as was the etch temperature of 1125°C. An HCl concentration of 0.42% for 10 minutes produced wafers which were free of haze and substantially free of stacking faults.

TABLE V

Effect of HCl Etch on Epitaxy Film Quality

(Standard Procedure as on pg 82 of 14874)

Run #/ Page	HCl Etch Flow Rate cc/min	HCl Etch Meter Setting	Time Min.	Resistivity Ω cm				Wafer Appearance
					188			
1/82	60	112	8	100	160	190	0.03289g	slight cloudiness on
(83A)					154.5			front. Monsanto wafer
2/83	60	112	8		229		0.03468g	cloudy all over no
(83B)				248	223	277		striation, heavier on
					157			front
3/83	60	112	8		540		0.03372g	cloudy all over
(83C)				236	250	182		heavier at front
					203			striations visible
4/84	60	112	8		284		0.02994g	cloudy all over
(84)				224	161	145		heavier at front
					161			striations visible
1/85	12	30	8		40		0.02139g	cloudy at front
(85A)				47	56	53		slightly stacking
					33			faults evident all over
2/85	12	30	5		62.9		0.02771g	stacking faults all
(85B)				45	54.3	48.8		over
					47			
3/85	60	112	5		75.4		0.02927g	cloudy at front
(85C)				38.6	75.7	77.4		striations visible
					70.9			stacking faults all
								over
4/86	25	50	5		349		0.02666g	very slight cloudiness
(86A)				75.5	95	116.7		at front-stacking
					81.9			faults all over
5/86	25	50	6		136.5		0.02468g	very slight cloudiness
(86B)				85.6	156.6	184.1		cloudy spot at front
					131.8			few stacking faults
1/87	25	50	6		102.4		0.02737g	slight cloudiness at
(87A)				91.5	96.9	144.9		front - some stacking
					86.6			faults
2/87	25	50	6		120.4		0.02785g	some cloudiness at
(87B)				157.8	107.2	135.7		front some stacking
					84.7			faults

TABLE V (Cont'd)

Run #/ Page	Flow Rate cc/min	Meter Setting	Time Min.	Resistivity Ω cm		Wafer Appearance
3/87 (87C)	25	50	6	63.9 40.1 52.0 62.6 47.8	0.02661g	Deposition at 1000°C Zone of thick cloudy deposition - remainder clear - few stacks
4/88 (88A)	25	50	10	184.6 156.6 157.0 164.6 124.9 Av 157.5	0.02939g	very very slight cloudiness at front stacking all over
5/88 (88D)	25	50	10	112.0 124.1 97.9 100.3 102.0 Av 107.3	0.02901g	wafer clean few stack
1/89 (89A)	25	50	10	242 439 399 401 783 Av 370	0.02687g	Clean wafer
2/89 (89B)	25	50	10	175 158 121 197 140 Av 158.2	0.02656g	Clean wafer - several stacks
3/89 (89C)	25	50	10	137 163 162 196 180 Av 167.6	0.02398g	Some cloudiness left side - very few stacks

There were slight resistivity variations across the wafer surface which are largely due to variations in film thickness. However, when five measurements are made at the positions shown in Figure 15, the average values are within 4% of the maximum variation. Higher resistivity values and thinner deposits were obtained on the initial test on any given day. This could be due to inadequate silane purge to remove the overnight nitrogen purge gas. This fact is significant for EPSDU operations where purging between samples will be very important for assuring representative results.

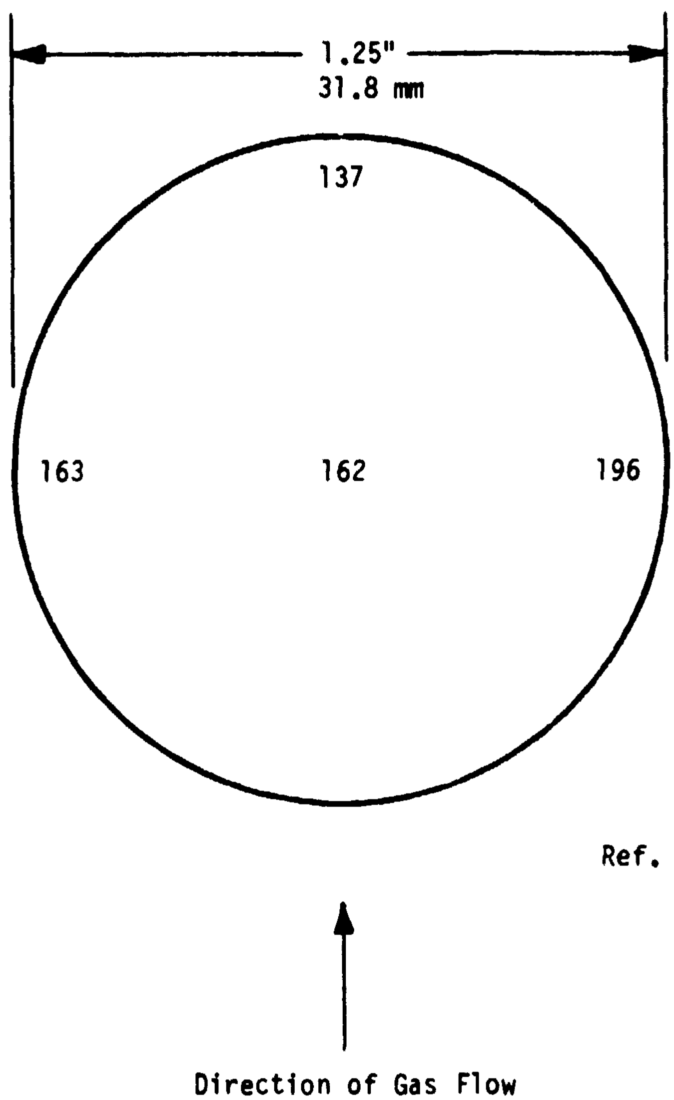
Epitaxial films of consistently high resistivity can be grown using a well-defined regime of precleaning and high temperature processing.

Two series of runs were made during December by adding controlled amounts of phosphine or diborane to silane. The purpose was to develop a correlation between impurity level in the sample silane and the resistivity of the deposited film.

For the case of phosphine, the deposits were made on P-type wafer substrates in order to form a N - P junction and permit measuring the epi-film resistivity. Figure 16 shows the result of those runs along with a plot of the theoretical resistivity assuming all of the phosphine had been incorporated.

A similar set of experiments was made using N-type wafer substrates. However, the baseline resistivity values using undoped silane ($\sim$165 Ωcm, N-type) were erratic, varying considerably from edge to edge across the wafer and also from wafer to wafer. This is not too surprising since relatively high-purity N-type silane was used as the control and thus, no junction was being made. One dopant run, using 17 ppb B_2H_6, resulted in a wide distribution of resistivity readings. The analysis of these deposits has not yet been completed, particularly the film thickness which significantly impacts the resistivity value.

A draft report detailing the epitaxy test method and results has been prepared. In spite of the erratic results with the diborane dopant run, the general method is felt to be valid for determining silane quality quickly and sufficiently accurate to meet EPSDU's needs.



Ref. N.B. 14874 P89C

Note:

1) Values in Ω cm

2) Average film thickness-15 μm
on 60 Ω cm P type substrate

Figure 15 Resistivity Variation with Position

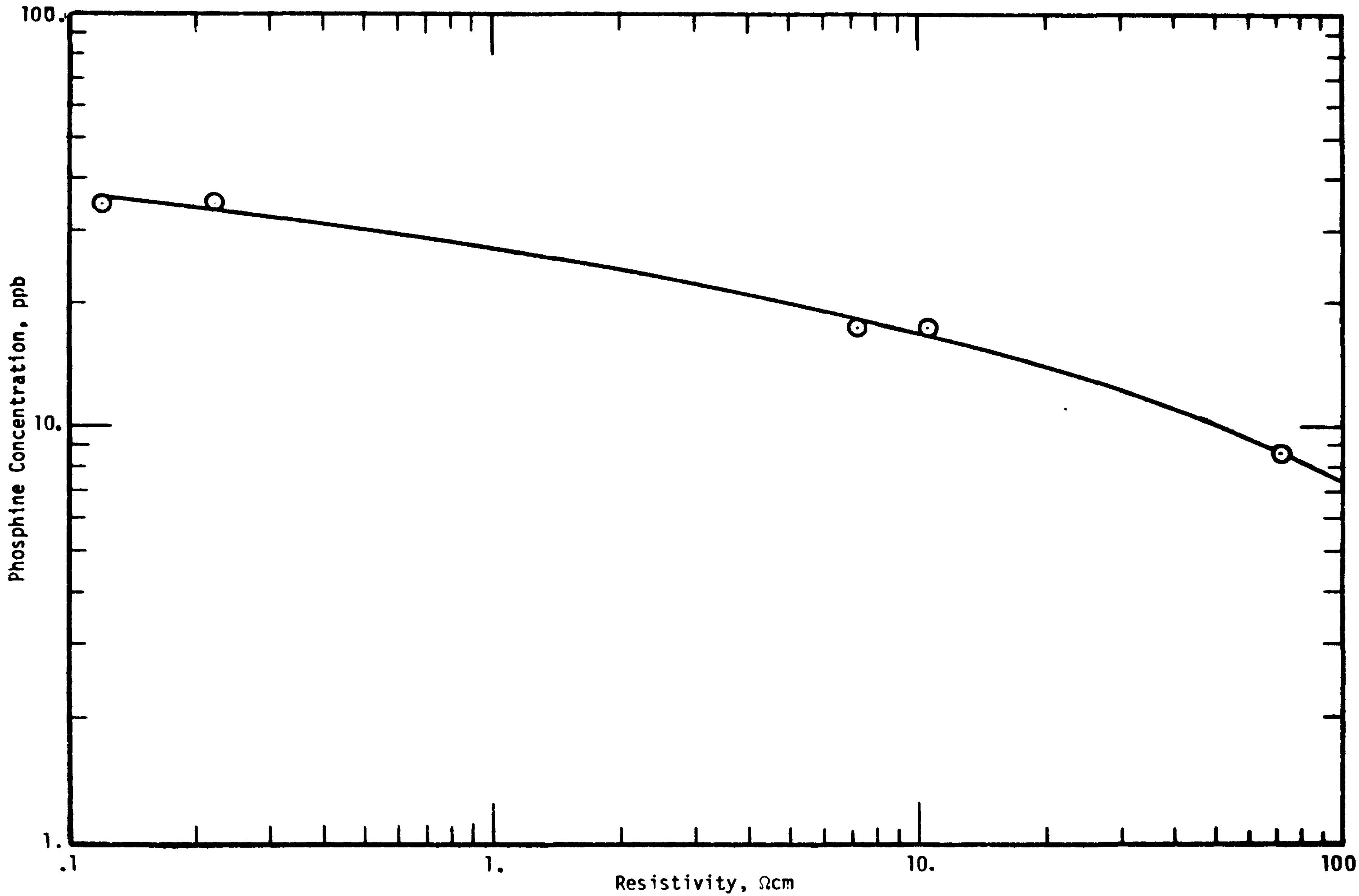


Figure 16 Resistivity vs Dopant Concentration - Silane Epitaxy on 60 Ωcm, P-type, Silicon Substrate

1.7 MANAGEMENT AND DELIVERABLES

This report item includes all activities associated with managing the program and insuring that all deliverables are made in accordance with the program requirements.

JPL/UCC Bi-Monthly Meeting

The JPL/UCC bi-monthly progress report meeting was held in Tonawanda in December. Side trips were made to East Chicago to witness the EPSDU civil work and to Rochester to observe the silicon shotter subcontract work at Kayex.

EPSDU Design Safety Review

The safety review meeting was held in November, 1980. An extensive review of the mechanical installation drawings was conducted in December which incorporated the results of the November safety review. The review revealed substantial changes which must be made from the standpoint of safety, operability, and cost. This type of additional work is normal and the expense is normally allocated as "contingency".

Quarterly Progress Review With Kayex

This review was conducted in December during the visit by UCC/JPL personnel to observe the shotter progress.

Funding for Fy1981

Development of various program options for Fy1981 was a major activity in November. Because of the funding constraint, planned activities which are not essential for EPSDU construction and start-up were dropped or reduced in scope. Essential activities were rescheduled to minimize expenditures in Fy1981.

Four program options were submitted to JPL for Fy1981 with one recommended for JPL approval. The Fy1981 expenditure schedule for this option was close to the funding limit, and the schedule slippage is minimized to 2 to 4 months.

Some personnel reduction was carried out in almost all areas in preparation for the program stretch out. In preparing the Fy1981 schedule and budget, out overriding emphasis is in minimizing the EPSDU installation delay. A substantial effort will be necessary in January 1981 to prepare a schedule and budget proposal to JPL, since any change in schedule and manpower requirements must be consistent with the overall Tonawanda work load for 1981 and beyond.

Major Milestones

The major milestones reached this quarter are presented in Table V.

TABLE V MAJOR MILESTONES REACHED

WBS NO.	MILESTONE	DESCRIPTION OF MAJOR MILESTONE
1.1.2.3	C	Scale Model Complete.
1.1.3.4	H	Process Equipment - Waste Treatment Equipment RFR's Issued.
1.1.3.8	K	Data Collection System - Equipment RFR's Issued.
1.2.1.1	D	Field Instruments and Controls - Quarterly Procurement Status Report Issued.
1.2.1.2	D	Panels and Panel Instruments - Quarterly Procurement Status Report Issued and All Equipment Received.
1.2.1.3	D	Data Collection System - Quarterly Procurement Status Report Issued.
1.2.1.4	D	Other Process Control Equipment - Quarterly Procurement Status Report Issued.
1.2.2.1	D,G	Power Supply - Quarterly Procurement Status Report Issued and All Equipment Received.
1.2.2.2	D	Power Control Center - Quarterly Procurement Status Report Issued.
1.2.2.3	D	Other Power System Equipment - Quarterly Procurement Status Report Issued.
1.2.3.2	D	Reactors - Quarterly Procurement Status Report Issued.
1.2.3.3	D	Distillation Columns - Quarterly Procurement Status Report Issued.
1.2.4.1	D	Compressors - Quarterly Procurement Status Report Issued.

TABLE V MAJOR MILESTONES REACHED (continued)

WBS NO.	MILESTONE	DESCRIPTION OF MAJOR MILESTONE
1.2.4.2	D	Pumps - Quarterly Procurement Status Report Issued.
1.2.4.3	D	Heat Exchangers - Quarterly Procurement Status Report Issued.
1.2.4.4	D	Tanks - Quarterly Procurement Status Report Issued.
1.2.4.5	D	Solids Handling - Quarterly Procurement Status Report Issued.
1.2.4.6	D	Waste Treatment - Quarterly Procurement Status Report Issued.
1.2.4.7	D	Other Process Equipment - Quarterly Procurement Status Report Issued.
1.2.5.1	D	Therminol Heater - Quarterly Procurement Status Report Issued.
1.2.5.2	D	Refrigeration Unit - Quarterly Procurement Status Report Issued.
1.2.5.3	D	Cooling Tower - Quarterly Procurement Status Report Issued.
1.2.5.4	D	Instrument Air Unit - Quarterly Procurement Status Report Issued.
1.2.5.6	D	Other Auxiliary Equipment - Quarterly Procurement Status Report Issued.
1.2.6.1	D	Q.C. Trailer - Quarterly Procurement Status Report Issued.
1.3.1.3	D	Structural - Start Structural Work.
1.6.1.1.7	H	Purity & Operability Tests - Consecutive Runs and 12-Hour Duration Runs Complete.
1.6.4.1.4	H,K	Method Development - Dopant Test Series No.1 Complete.
1.6.4.3.4	L	Method Development - Silane Analysis - Epitaxy Report Draft Issued.
1.7.3	K,L,M	Monthly Financial and Management Reports.
	K,L,M	Monthly Technical Progress Reports
	E,F	Bi-Monthly Review Meeting
	D	Quarterly Technical Progress Report for the Third Quarter 1980 Issued.

SECTION I CONCLUSIONS

Significant highlights and conclusions are presented to the relevant WBS numbers.

1.1 DESIGN/PROCUREMENT

1.1.1 Process Design

- Detailed reviews of drawings were conducted to assure that they were compatible with the P&I diagrams, installation drawings and the scale model.
- Designs were thoroughly reviewed from operability and safety points of view.
- Equipment cleaning requirements were established.
- Valves including pressure relief valves were selected.

1.1.2 Facility Design

- The safety review team met and made comments which were implemented into the EPSDU design.
- The gantry scale model was completed.
- The facility scale model equipment was completed and ready for assembly according to the EPSDU plot plan.
- Operations liaison was instituted and the plant manager is becoming familiar with design and procurement.

1.1.3 Equipment Design, Specification, Procurement

- The process control system design was 'frozen'.
- The P&ID was updated to collect all changes.
- The main control panel design was completed and issued for quotation.

- Silica agglomerator drawings and specifications were completed and RFQ issued.
- Data col'ection system design is progressing.

1.1.4 Installation Design, Specification, Procurement

- Initial draft of mechanical design package was completed.
- EPSDU desig.. review was completed in December. Major changes to the mechanical design package resulted in additional design and drafting activity to improve operability, maintenance and reduce piping installation costs.
- The electrical d sign effort is continuing and will be updated to be compatible with mechanical design changes.

1.2 EQUIPMENT FABRICATION/DELIVERY

- Many purchase orders were issued for instrumentation and panel controls.
- Major process equipment (UCC supplied) - reactor, distillation columns, will be shipped in first quarter of 1981.
- Vendor supplied equipment - heat exchangers, pumps etc. are due to be shipped in January 1981.
- QC Trailer is on order for delivery in xt quarter.
- Inspections at vendor plants have taken place to ensure conformance to specifications for custom process equipment.

1.3 INSTALLATION AND CHECKOUT

- Civil, underground and structural work is near completion.
- Major equipment items will be delivered to EPSDU site in January.
- Valves and associated longer lead items have been ordered.

1.6 PROCESS SUPPORT R&D

1.6.1 Free-Space Reactor Development

- Theoretical analyses have taken place to model the reactor for subsequent scale-up.
- Experimental verification of the reactor model will be issued as a report.
- Test results of silane conversion runs were reported.
- A 12-hour duration run was accomplished with excellent results.

1.6.2 Melting/Consolidation

- The basic design of the shotting and melting system was completed and parts were placed on order.
- The shotter framework and platform was installed and the assembly of parts were installed upon receipt.
- Successful powder melting and crystal pulling was demonstrated.

1.6.3 Fluid-Bed Development

- The PDU design was completed in October 1980.
- An operations procedure was written.
- Construction and instrumentation was completed.

1.6.4 Quality Control

- Polycrystalline silicon runs were grown in which high purity silane gas was used, to establish a standard.
- Other growth cycles were made in which phosphine and diborane dopant gases were introduced (levels from 2 to 20 ppb).

SECTION IV PROJECTED QUARTERLY ACTIVITIES

1.1 EPSDU DESIGN/PROCUREMENT

1.1.1 Process Design

- Review mechanical installation drawings.
- Review of logic for startup/shutdown.
- Complete specification of safety relief valves.
- Assemble process pyrolysis design package.

1.1.2 Facility Design

- Conduct safety reviews of all systems.

1.1.3 Equipment Design, Specification, Procurement

- Issue final P&ID.
- Issue equipment purchase orders.
- Continue programming of data collection systems.
- Assembly of specialty items.

1.1.4 Installation Design, Specification, Subcontract

- Issue RFR for piping components.
- Obtain quotations for silane tanks.
- Continue mechanical drawing activity.
- Continue electrical drawing activity.

1.2 EQUIPMENT FABRICATION/DELIVERY

- Issue validated purchase orders for all equipment items.
- Issue monthly and quarterly procurement status reports.

- Continue processing and review of vendor drawing information.
- Issue applicable purchase order change notices for equipment design changes identified during vendor drawing reviews.
- Visit fabricators of custom process equipment.
- Initiate thorough inspection activity at EPSDU.
- Review equipment cleaning procedures.

1.3 INSTALLATION & CHECKOUT

- Complete civil subcontract work.
- Issue field progress reports.
- Set large pieces of equipment as received at EPSDU.

1.4 OPERATION

- Plant manager will become familiar with process and facility during construction design.
- Update utility and raw material costs and develop detailed plan for Fy1981.
- Establish equipment inspection procedures and initiate inspection of equipment received.

1.5 COMMERCIAL PROCESS ECONOMIC ANALYSIS

(No activity)

1.6 PROCESS SUPPORT R&D

1.6.1 Free-Space Reactor Experiments

- Prepare final report on PDU experiments.
- Shutdown and secured.
- Obtain analytical data on silicon powder.

1.6.2 Melting Consolidation

- Check out induction heater.
- Complete assembly and checkout of melter system.
- Attempt an initial melting run.
- Commission the induction generator.
- Prepare preliminary design specifications for melter/shotter.

1.6.3 Fluid-Bed Development

- Complete PDU installation.
- Complete checkout of PDU.
- Conduct safety review.
- Calibrate silane and hydrogen flowmeters.
- Set up gas chromatograph and oxygen analyzer.

1.6.4 Quality Control

- Complete final report for slim-rod and epitaxy reactor development.

1.7 MANAGEMENT AND DELIVERABLES

- Prepare cost back-up materials requested by JPL.
- Update Phase III program status and estimate to complete.

APPENDIX A

EQUIPMENT PROCUREMENT STATUS

DECEMBER 1980

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
453-04, 05 Waste Gas Induction Blower	50001	✓	✓	✓	✓	✓	✓	
453-02 Agglomeration Blower	50002	✓	✓	✓	✓	✓	✓	
	50003							
	50004							
426-02 Quench Contactor Pump	50005	✓	✓	✓	✓	✓	✓	
	50006							
	50007							
	50008							
	50009							
423-02, 03 Recycle H_2 Compressor	50010	✓	✓	✓	✓	✓	✓	
443-02 Pyrolysis H_2 Compressor	50011	✓	✓	✓	✓	✓		
466-02 Hot Oil Pump	50012	✓	✓	✓	✓	✓		
466-04, 05 Cooling Water Pump	50013	✓	✓	✓	✓	✓	✓	
424-02 Quench Condenser	50014	✓	✓	✓	✓	✓	✓	
[illegible]-04 [illegible]-06, 10 Reboilers	50015	✓	✓	✓	✓	✓		

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
434-02 Stripper Condenser	50016	✓	✓	✓	✓	✓	✓	
421-12, 16 441-06 Tanks	50017	✓	✓	✓	✓	✓	✓	
464-02, 04 Ventilation Heat Exchaners	50018							
434-08, 14, 18 Column Condensers	50019	✓	✓	✓	✓	✓	✓	
	50020							
	50021							
434-12, 16, 24 444-02 Coolers	50022	✓	✓	✓	✓	✓	✓	
	50023							
	50024							
	50025							
	50026							
	50027							
434-26 Refrig. Heating Coil	50028							
	50029							
	50030							

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
	50031							
	50032							
411-02, 441-04 461-08 Bins	50033	✓	✓	✓	✓	✓		
	50034							
421-02,04,06,08,10,14,18 451-04,06 Tanks	50035	✓	✓	✓	✓	✓		
	50036							
	50037							
	50038							
	50039							
431-04, 06, 08, 10 435-02, 04 Tanks & Reactors	50040	✓	✓	✓	✓	✓	✓	
	50041							
	50042							
	50043							
	50044							
	50045							

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
	50046							
	50047							
	50048							
	50049							
	50050							
451-10 Waste Neutralizer	50051							
	50052							
	50053							
	50054							
461-02 Hot Oil Expansion Tank	50055	✓	✓	✓	✓	✓		
425-02 Hydrogenation Reactor	50056	✓	✓	✓	✓	✓	✓	
445-02 Quartz Liner	50057							
	50058							
443-02 Pyrolysis Reactor and Hopper	50059							
417-02 457-04, 06 Filters	50060	✓	✓	✓	✓	✓		

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
427-02 Crude TCS Filter	50061	✓	✓	✓	✓	✓	✓	
437-02 Silane Ultra Filter	50062	✓	✓	✓	✓	✓		
	50063							
	50064							
	50065							
	50066							
448-08, 10 Loading Scales	50067							
	50068							
448-04 Boule Cart	50069							
448-14 Boule Scale	50070							
	50071							
	50072							
458-04 Silica Drum Packer	50073	✓	✓	✓	✓	✓		
	50074							

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
459-02, 04 Venturi and Scrubber	50075	✓	✓	✓	✓	✓	✓	
429-04 Superheater	50076	✓	✓	✓	✓	✓		
	50077							
	50078							
	50079							
	50080							
	50081							
	50082							
459-08, 10, 12, 14 Waste Burners	50083	✓	✓	✓	✓	✓		
	50084							
454-04 Silica Agglomerator	50085							
469-02 Cooling Tower	50086	✓	✓	✓	✓	✓	✓	
469-06 Cooling Tower Treatment	50087	✓	✓	✓	✓	✓	✓	
469-12 Refrigeration System	50088	✓	✓	✓	✓	✓	✓	

C-2

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIPMENT REC'D
469-16 Therminol Heater	50089	✓	✓	✓	✓	✓		
452-02 Muriatic Tailing Column	50090	✓	✓	✓	✓	✓		
456-08 Tailing Column Pump	50091	✓	✓	✓	✓	✓	✓	
469-14 In trument Air Package	50092	✓	✓	✓	✓	✓	✓	
642-02 MCC	50093	✓	✓	✓	✓	✓		
641-02 Transformer	50094	✓	✓	✓	✓	✓	✓	
	50095							
365-02 Quality Control Trailer	50096	✓	✓	✓	✓	✓		
	50097							
411-08 TL Argon Tank	50098							
461-04, 06 Fuel Oil Storage Tank	50099	✓	✓	✓	✓	✓		
	50100							
	50101							
643 Emergency Generator	50102	✓	✓	✓	✓	✓		

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
463-06, 08 Ventilation Blower	50103							
	50104							
426-06, 07 STC Pumps	50105	✓	✓	✓	✓	✓	✓	
	50106							
For 432-08 Internals for Silane Column	50107	✓	✓	✓	✓	✓	✓	✓
Chlorosilane Analysis	50108	✓	✓	✓	✓	✓		
UV Spectrophotometer	50109	✓	✓	✓	✓	✓		✓
Elemental Analysis	50110	✓	✓	✓	✓	✓		✓
Silicon Melting Furnace	50111	✓	✓	✓	✓	✓		✓
432-02 Stripper Column	50112	✓	✓	✓	✓	✓		
432-04 TCS Column	50113	✓	✓	✓	✓	✓		
432-06 DCS Column	50114	✓	✓	✓	✓	✓		
459-16 Agitator	50115	✓	✓	✓	✓	✓	✓	
429-02 Quench & Solids Removal Contractor	50116	✓	✓	✓	✓	✓	✓	

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
	50117							
	50118							
466-06, 07 Fue' Oil Pumps	50119	✓	✓	✓	✓	✓		
436-04 DCS Distillate Pump	50120	✓	✓	✓	✓	✓	✓	
	50121							
432-08 Silane Column	50122	✓	✓	✓	✓	✓		
	50123							
Field Instrumentation	50124	✓	✓	✓	✓	✓		
469-20 Instrument Air Dryer	50125	✓	✓	✓	✓	✓	✓	
428-04 Solids Conveyor	50126	✓	✓	✓	✓	✓	✓	
	50127							
	50128							
464-06 Thaw Heater	50129	✓	✓	✓	✓	✓	✓	
Program Controller	50130	✓	✓	✓	✓	✓		

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
7 Haskel Pumps	50131	✓	✓	✓	✓	✓		
Pressure Lubricators	50132	✓	✓	✓	✓	✓		✓
8 BP Valves & 7 Springs	50133	✓	✓	✓	✓	✓		✓
	50134							
Printer	50135	✓	✓	✓	✓	✓		
Computer	50136	✓	✓	✓	✓	✓		
Memorex Cartridges	50137	✓	✓	✓	✓	✓		
Automatic Valves	50138	✓	✓	✓	✓	✓		
Automatic Valves	50139	✓	✓	✓	✓	✓		
Automatic Valves	50140	✓	✓	✓	✓	✓		
Augomatic Valves	50141	✓	✓	✓	✓	✓		
Automatic Valves	50142	✓	✓	✓	✓	✓		
	50143							
	50144							
7 Gauges	50145	✓	✓	✓	✓	✓		✓

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	CERTIFIED DWG REC'D	EQUIP-MENT REC'D
Pressure Lubricators	50146	✓	✓	✓	✓	✓		✓
Thermocouples	50147	✓	✓	✓	✓	✓		
7 Level Switches	50148	✓	✓	✓	✓	✓		
	50149							
Sewer Tie-In Line	50150	✓	✓	✓	✓	✓		
Service Agreement	50151	✓	✓	✓	✓	✓		
	50152							
Lever Transmitters & Switches	50153	✓	✓	✓	✓	✓		
Field Instrumentation	50154	✓	✓	✓	✓	✓		
Conditioners & Process Filters	50155	✓	✓	✓	✓	✓		
Sample Conditioners	50156	✓	✓	✓	✓	✓		
Sample Conditioners	50157	✓	✓	✓	✓	✓		
	50158							
	50159							
Caustic Storage Tank	50160	✓	✓	✓	✓	✓		

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
Immersion Heater	50161	✓	✓	✓	✓	✓		
Fuel Tank	50162	✓	✓	✓	✓	✓		
Shipping Preparation & Freight	50163					✓		
Automatic Valve	50164	✓	✓	✓	✓	✓		
	50165							
Main Instrument Panel "A"	50166	✓	✓	✓	✓	✓		
Main Instrument Panel "A"	50167	✓	✓	✓	✓	✓		
Field Instrumentation	50168	✓	✓	✓	✓	✓		
Main Instrument Panel "A"	50169	✓	✓	✓	✓	✓		
Main Instrument Panel "A"	50170	✓	✓	✓	✓	✓		
	50171							
Main Instrument Panel "A"	50172							
Main Instrument Panel "A"	50173	✓	✓	✓	✓	✓		
Main Instrument Panel "A"	50174							
Main Instrument Panel "A"	50175	✓	✓	✓	✓	✓		

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
Main Instrument Panel "A"	50176	✓	✓	✓	✓	✓		
Field Instrumentation	50177	✓	✓	✓	✓	✓		
Field Instrumentation	50178	✓	✓	✓	✓	✓		
Field Instrumentation	50179	✓	✓	✓	✓	✓		
Field Instrumentation	50180	✓	✓	✓	✓	✓		
Field Instrumentation	50181	✓	✓	✓	✓	✓		
Field Instrumentation	50182	✓	✓	✓	✓	✓		
Field Instrumentation	50183	✓	✓	✓	✓	✓		
Field Instrumentation	50184	✓	✓	✓	✓	✓		
Field Instrumentation	50185	✓	✓	✓	✓	✓		
	50186							
Filter Bags	50187	✓	✓	✓	✓	✓		
Valves	50188	✓	✓	✓	✓	✓		
Valves	50189	✓	✓	✓	✓	✓		
Automatic Valves	50190	✓	✓	✓	✓	✓		

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIP-MENT REC'D
Automatic Valves	50191	✓	✓	✓	✓	✓		
Automatic Valves	50192	✓	✓	✓	✓	✓		
Automatic Valves	50193	✓	✓	✓	✓	✓		
Check Valves	50194	✓	✓	✓	✓	✓		
	50195							
	50196							
Field Instrumentation	50197	✓	✓	✓	✓	✓		
	50198							
Field Instrumentation	50199	✓	✓	✓	✓	✓		
Field Instrumentation	50200	✓	✓	✓	✓	✓		
Field Instrumentation	50201	✓	✓	✓	✓	✓		
Controller	50202	✓	✓	✓	✓	✓		
	50203							
	50204							
Instrument Field List	50205	✓	✓	✓	✓	✓		

EQUIPMENT PROCUREMENT STATUS

EQUIPMENT NO. & NAME	P. O. NUMBER	FUNC SPECS ISSUED	ENG SPECS ISSUED	RFQ ISSUED	RFR ISSUED	P.O. ISSUED	DWG REC'D	EQUIPMENT REC'D
	50206							
Rubber Hose	50207					✓		
	50208							
	50209							
	50210							
	50211							
Field Instrumentation	50212	✓	✓	✓	✓	✓		
	50213							
Field Instrumentation	50214	✓	✓	✓	✓	✓		
Field Instrumentation	50215	✓	✓	✓	✓	✓		
	50216							